

UNCLASSIFIED

AD NUMBER
AD867012
NEW LIMITATION CHANGE
TO Approved for public release, distribution unlimited
FROM Distribution authorized to U.S. Gov't. agencies and their contractors; Administrative/Operational use; Mar 1970. Other requests shall be referred to Air Force Materials Laboratory [MAAE], Wright-Patterson Air Force Base, Ohio 45433.
AUTHORITY
AFSC, USAF ltr, 2 Mar 1972.

THIS PAGE IS UNCLASSIFIED

AFML-TR-69-319

AD 867012

EVALUATION OF HIGH TEMPERATURE FIRE AND EXPLOSION SUPPRESSANT CONFIGURATIONS FOR AIRCRAFT FUEL SYSTEM APPLICATIONS

G.L. Ball III

A. Wojtowicz

I.O. Salyer

MONSANTO RESEARCH CORPORATION

Technical Report AFML-TR-69-319

March 1970

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory (MAAE) Wright-Patterson Air Force Base, Ohio 45433

**AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO**

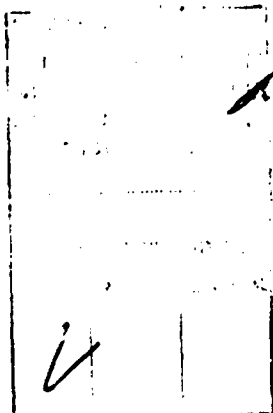
NOTICES

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government would thereby encourage no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by any implication otherwise as in any manner licensing the holder for any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented inventions that may in any way be related thereto.

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory (MAAE) Wright-Patterson Air Force Base, Ohio 45433.

The distribution of this report is limited because it relates to vulnerability of aircraft to ground fire.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.



EVALUATION OF HIGH TEMPERATURE FIRE AND EXPLOSION
SUPPRESSANT CONFIGURATIONS FOR AIRCRAFT FUEL SYSTEM APPLICATIONS

G. L. Ball III

A. Wojtowicz

I. O. Salyer

March 1970

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory (MAAE) Wright-Patterson Air Force Base, Ohio 45433.

FOREWORD

This report covers work done by Monsanto Research Corporation, Dayton Laboratory, Dayton, Ohio, under Contract F33615-67-C-1716, from 1 July 1968 to 30 September 1969. This research was initiated under Project No. 7381 "Materials Applications", Task No. 738108 "Application of Materials and Processes for Limited War Support".

The program was administered under the direction of the Air Force Materials Laboratory with E. J. Morrissey as Project Engineer. For Monsanto Research Corporation, G. L. Ball III served as Project Leader in the Polymer Applications Section under I. O. Salyer, Manager. The Principal Investigator on the project was Mr. A. Wojtowicz, who was assisted by Mr. R. D. Myers.

The contractor's report number is MRC DA 239.

The report was submitted by the authors in November 1969.

This technical report has been reviewed and is approved.

Albert Olevitch

ALBERT OLEVITCH, Chief
Materials Engineering Branch
Materials Support Division
Air Force Materials Laboratory

ABSTRACT

A program was conducted for investigation of materials to provide improved thermal resistance in an aviation fuel environment. It was desirable that the compositions obtained be similar to a 10-pore-per-inch reticulated urethane foam used in a fire and explosion suppressant application.

Toward providing polymeric structures with improved thermal resistance, four classes of materials were investigated. These were coated foams, uncoated foams, filled thermoplastics, and unfilled thermoplastics. Specific materials were selected and evaluated for their retention of mechanical performance as a function of time following exposure to JP-5 fuel at elevated temperatures.

The most promising materials obtained in the program consisted of a polyester fiber mat and a low-density, open-celled polyimide foam. Both of these materials exhibited excellent retention of mechanical strength and modulus for 10 week exposure periods in JP-5 fuel at 325°F.

Foams coated with nylon 11, thermoplastic polyimide, and polymer 380 all exhibited some improvement in mechanical performance over their substrate reticulated urethane foam. However, none of these materials proved even partially adequate for the refluxing JP-5 fuel environment.

Of the thermoplastics investigated, the nylon 66, polyimide, and polymer 360 all exhibited a substantial retention of mechanical properties in the hyperthermal fuel environment. Of importance was the inclusion of glass fillers, which provided geometrical integrity to the thermoplastic while at the elevated temperatures.

In order to establish some utility for the coated urethane foams the mechanical properties of a series of foams were evaluated at temperatures less than 325°F and greater than ambient and a useful temperature of 200°F was established.

(This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory (MAAE), Wright-Patterson Air Force Base, Ohio 45433).

TABLE OF CONTENTS

	<u>Page</u>
<u>SECTION I</u>	
INTRODUCTION	1
<u>SECTION II</u>	
SUMMARY AND CONCLUSIONS	2
<u>SECTION III</u>	
EXPERIMENTAL DISCUSSION	4
1. EXPOSURE AND TESTING TECHNIQUES	4
a. Phase 1 (Continuous) Exposure	4
b. Phase 2 (Thermal cyclic) Exposure	4
c. Physical Testing Techniques	5
2. THERMAL-RESISTANT FOAMS	6
a. Coated Foams	6
b. Uncoated Foams	8
3. THERMAL-RESISTANT THERMOPLASTICS	9
a. Filled Thermoplastics	10
b. Unfilled Thermoplastics	11
APPENDIX - Table and Figures	12

Preceding Page Blank

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Foam Type Structures Which Showed Promise for Use at 325°F in JP-5 Fuel for Extended Periods of Time.	17
2	Environmental Baths Used to Expose Candidate Materials in JP-5 Fuel at Temperatures up to 325°F.	18
3	Time-Temperature Sequence for Phase 2 Type Thermocycling.	19
4	Test Configurations and Arrangements Used to Characterize Candidate Foam and Thermoplastic Materials.	20
5	Photograph Illustrating the Modulus Characteristics of the Coated and Uncoated Foams.	21
6	Stress vs Compression for Nylon 11 Single Coated Urethane Foam Following Phase 1 Exposure for Four Weeks.	22
7	Stress vs Compression for Nylon 11 Double Coated Urethane Foam Following Phase 1 Exposure for Four Weeks.	23
8	Stress vs Compression for Reticulated Urethane Foam Coated with Polyimide Following Phase 1 Exposure for One Week.	24
9	Stress vs Compression for Reticulated Urethane Foam Coated with Polymer 380 Following Phase 1 Exposure for Four Weeks.	25
10	Photographs of Coated Urethane Foams Following Exposure to JP-5 Fuel at Elevated Temperatures.	26
11	Stress vs Compression for Reticulated Urethane Foam Coated with Nylon 11 Following Exposure at 200°F in JP-5 Fuel for Four Weeks.	27
12	Stress vs Compression for Reticulated Urethane Foam Coated with Nylon 11 Following Exposure at 250°F in JP-5 Fuel for Four Weeks.	28
13	Stress vs Compression for Reticulated Urethane Foam Coated with Polyimide Following Exposure at 200°F in JP-5 Fuel for Four Weeks.	29
14	Stress vs Compression for Reticulated Urethane Foam Coated with Polyimide Following Exposure at 250°F in JP-5 Fuel for Four Weeks.	30
15	Stress vs Time of Exposure and Exposure Temperature for Reticulated Urethane Foam Coated with Nylon 11.	31

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
16	Stress vs Time of Exposure and Exposure Temperature for Reticulated Urethane Foam Coated with Polyimide.	32
17	Stress vs Compression for Nylon 11-Coated Urethane Foam Following Phase 2 Thermocycling for 100 Cycles (ten weeks).	33
18	Compressive Stress as a Function of Exposure Time to Phase 2 Thermocycling for Nylon 11-Coated Urethane Foam for 100 cycles (ten weeks).	34
19	Thermal Gravimetric Analysis of the Uncoated and Coated Urethane Foams in Air.	35
20	Thermal Gravimetric Analysis of the Urethane and Coated Urethane Foams in an Inert Atmosphere.	36
21	Stress vs Compression for Untreated Reticulated Urethane Foam Following Exposure at 325°F in JP-5 Fuel for Five Days.	37
22	Stress vs Compression for Uncoated Reticulated Urethane Foam Following Exposure at 200°F in JP-5 Fuel for Four Weeks.	38
23	Stress vs Compression for Uncoated Reticulated Urethane Foam Following Exposure at 250°F in JP-5 Fuel for Four Weeks.	39
24	Reticulated Urethane Foam Following One Week Immersion in UP-5 Fuel at 325°F (Phase 1 Exposure).	40
25	Photographs of Uncoated Urethane Foams Following Exposure to UP-5 Fuel at Elevated Temperatures.	41
26	Stress vs Compression for Uncoated Reticulated Urethane Foam Following Phase 2 Thermocycling for 40 Cycles.	42
27	Stress vs Compression of Polyester Fiber Foam Following Phase 1 Exposure for Four Weeks.	43
28	Stress vs Compression of Polyester Fiber Foam Following Phase 2 Exposure for 100 Cycles (ten weeks).	44
29	Photographs of High Temperature Foams Following Exposure to JP-5 Fuel at Elevated Temperatures.	45

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
30	Stress vs Compression for Polyimide Foam Following Phase 1 Exposure for Four Weeks.	46
31	Stress vs Compression for Polyimide Foam Following Phase 1 Exposure for Ten Weeks.	47
32	Thermal Gravimetric Analysis of the Polyester and Polyimide Foams in Air.	48
33	Thermal Gravimetric Analysis of the Polyester and Polyimide Foams in an Inert Atmosphere.	49
34	Large Celled Polyimide Foam Prepared From Polyimide Powders by Salt Ball Extraction Technique (Density 2.4 lb/ft ³).	50
35	Stress vs Compression for Large Celled Polyimide Foams Following Phase 1 Exposure for Four Weeks.	51
36	Stress vs Compression for Large Celled Polyimide Foams Following Phase 2 Exposure for 100 Cycles (ten weeks).	52
37	Tensile Stress vs Strain for Glass Filled Nylon 66 Following Phase 1 Exposure for Four Weeks.	53
38	Tensile Stress vs Strain for Glass Filled Nylon 66 Following Phase 1 Exposure for Eight Weeks.	54
39	Tensile Stress vs Strain for Glass Filled Polyester Following Phase 1 Exposure for Four Weeks.	55
40	Tensile Stress vs Strain for Unexposed Glass Filled Polychlorotrifluoroethylene.	56
41	Tensile Stress vs Strain for Unexposed Polyvinylidene-chloride.	57
42	Photographs of Glass Filled Thermoplastics Following Exposure to JP-5 Fuel at Elevated Temperatures.	58
43	Photographs of Glass Filled Thermoplastics Following Exposure to JP-5 Fuel at Elevated Temperatures.	59
44	Tensile Stress vs Strain for Polymer 360 Following Phase 1 Exposure for Four Weeks.	60
45	Tensile Stress vs Strain for Thermoplastic Polyimide Following Phase 1 Exposure for Four Weeks.	61
46	Photographs of Thermoplastic Materials Following Exposure to JP-5 Fuel at Elevated Temperatures.	62

SECTION I

INTRODUCTION

The feasibility of utilizing reticulated urethane foam and perforated hollow spheres as fire and explosion suppressant materials in aircraft fuel tanks has been demonstrated by the U. S. Air Force. While the performance of a .10-pore-per-inch reticulated urethane foam was excellent, the efficacy of the perforated hollow spheres was shown to be considerably less.

The reticulated urethane foam had been extensively investigated and its applicability in aircraft fuel systems over the temperature range from -65 to +165°F was established. The foam was and is being used by the U. S. Air Force in aircraft fuel tanks for fire and explosion suppression.

Flight temperature profiles exceeding +165°F existed. Therefore, the Air Force developed a requirement for a similar foam system that would be usable and stable at temperatures up to 325°F in jet fuel. The approaches suggested were to provide the temperature resistance through the addition of coatings to the reticulated urethane foam system or to provide high temperature foam systems *per se*. The effectiveness of the coated urethane was contingent upon the lack of effect of the degradation products of the urethane at 325°F on the coated structure.

Since it was possible that the coated urethanes could retain their mechanical performance at temperatures greater than that of the uncoated urethane but somewhat below the 325°F temperature, they were also evaluated on a selective basis at 200° and 250°F.

It has been shown that the hollow perforated spheres which had some limited utility in the fire and explosion suppressant applications could be formed by injection molding followed by heat sealing. Therefore, thermoplastic materials that could be injection molded and also provide the thermal resistance in the fuel environment were investigated. It was only during the performance of this program that the limited utility of the hollow perforated spheres was established and therefore full effort was abandoned.

Accordingly the program involved the preparation of various coated and uncoated foams, and filled and unfilled thermoplastics in the form of compression and tensile test specimens; exposure to the various thermal environments in JP-5 fuel; followed by mechanical testing of the specimens at specific times or after a given number of cycles. Thermal gravimetric analysis was also conducted on the coated foams in order to demonstrate the effect of the various coatings on the thermal performance of the urethane.

SECTION II

SUMMARY AND CONCLUSIONS

The materials investigated for potential stability in a hyperthermal fuel environment fell into four primary categories. These were:

- Coated foams,
- Uncoated foams,
- Filled thermoplastics, and
- Unfilled thermoplastics.

The materials were subjected to an environmental exposure of complete immersion in JP-5 fuel at elevated temperatures. Two different thermal profiles were used. They comprised Phase 1, a more severe screening test, and Phase 2, a representative long-term exposure. Phase 1 exposure consisted of a continued submersion at 325°F for seven-day periods. Phase 2 consisted of representative flight temperature profiles from ambient up to 325°F. The Phase 2 profile included stepped temperature increments over test periods consisting of seven days, of which five were used to obtain 10 thermal cycles. Resistance to the exposure was judged by the retention of mechanical stress-strain characteristics.

The coated foams all had a substrate of the Scott Paper Company 10-pore-per-inch reticulated urethane foam. The coatings included Nylon 11, a thermoplastic polyimide, and polymer 380, which were all applied by Scott Paper Company.

The uncoated foams investigated included the 10-pore-per-inch reticulated urethane, a polyester fiber mat and two types of polyimides.

The filled thermoplastics, all incorporating glass fibers, consisted of two nylon 66 products, a polyester, polychlorotrifluoroethylene, polyvinylidene-chloride, polycarbonate, and polytetrafluoroethylene.

The unfilled thermoplastics investigated included a thermoplastic polyimide, polymer 360, polysulfone, polyphenylene oxide, and nylon 66.

The uncoated foams, except the urethanes, by far outperformed the other materials and exhibited retention of mechanical properties for periods of 10 weeks both after continuous exposure in JP-5 fuel at 325°F and

thermal cyclic Phase 2 exposure. The best uncoated foams were the polyimide and the polyester fiber foams. The uncoated reticulated urethane by itself did not perform well and was degraded to less than one-half its original strength within one day. Modified exposure tests, however, indicated that the reticulated urethane would be useful at temperatures up to 200°F.

The coated urethane foams all exhibited insufficient retention of properties on exposure to JP-5 fuel at elevated temperatures to warrant their further consideration. In addition to this lack of retention of mechanical properties, the coated urethanes also had higher initial moduli than the original reticulated urethane foam.

Both filled and unfilled thermoplastics were investigated. The primary advantage of filling was to provide geometrical integrity during the exposure of the various materials. However, no added thermal stability was anticipated. Those materials which exhibited the best retention of mechanical properties upon exposure to JP-5 fuel at 325°F were polyimide, polymer 360, glass-filled nylon, and glass-filled polytetrafluoroethylene.

The most promising of all the materials were the polyimide and polyester fiber foams, which are shown in Figure 1 as they were initially and after exposure to JP-5 fuel at 325°F.

An inadvertent burning of the polyimide foams demonstrated that they were stable to much higher temperatures than 325°F for a short time. They were converted to carbon-like foams with reasonable structural integrity in the presence of burning fuel. While the exact temperatures were not known, the pyrex glass cylinder in which these specimens were contained was melted.

One additional advantage of both the polyimide foam and the polyester fiber foam was that their modulus characteristics were in the range of the original reticulated urethane foam. It was anticipated, therefore, that they would have a reasonable chance of functioning as a fire and explosive suppressant void filler in the fuel tanks of aircraft.

Thermal gravimetric analysis of the foams coated with the various materials indicated an improved retention of weight as a function of temperature up to 1000°C. The materials in the order of added thermal resistance were: Nylon 11, polyimide, and polymer 380. The polyimide and polyester fiber foams, however, retained their weight and integrity at temperatures well beyond the coated reticulated urethanes. The polyimide foam also exceeded the polyester fiber foam in thermal performance.

SECTION III

EXPERIMENTAL DISCUSSION

The experimental discussion is quite straight-forward since the program consisted of selecting, obtaining, or preparing; thermal exposure; and mechanical testing of various materials. The discussion is subdivided into the exposure and testing techniques, thermal-resistant foams, and thermal-resistant thermoplastics.

1. EXPOSURE AND TESTING TECHNIQUES

The exposures used all involved the complete submersion of test specimens in JP-5 fuel at various elevated temperatures. The two types of exposures were referred to as Phase 1 type and Phase 2 type. The Phase 1 type consisted of isothermal exposures at specific temperatures and the Phase 2 type of thermal cyclic exposure. The testing techniques involved mechanical evaluation, subjective evaluations based on observations, and thermal gravimetric analysis.

a. Phase 1 (Continuous) Exposure

Phase 1 exposure consisted of a continuous and complete immersion of the test specimen in JP-5 fuel refluxing at 325°F. To a limited extent, a modified Phase 1 exposure was included which incorporated temperatures of 200 and 250°F.

The exposure was conducted in glass flasks with condensers to retain as much of the refluxing fuel as possible. The flask and the JP-5 fuel were heated by immersion of the flask in an oil bath maintained at the desired temperature. A photograph of this setup is shown in Figure 2.

The Phase 1 cycle was a continuous exposure over a seven-day week at the specified temperature.

b. Phase 2 (Thermal cyclic) Exposure

Phase 2 exposure was set up to simulate a flight temperature profile in JP-5 fuel. The exposure was conducted in a manner identical to the Phase 1 exposure with the exception that the specimen-filled flasks were transferred in their entirety to the various temperature oil baths for specific periods of time (see Figure 2).

The test cycle was set up to provide a temperature profile which represented two flights per day (or cycles), and an overnight exposure at ambient. The particular exposure conditions were:

- (a) 30 minutes at 150°F
- (b) 20 minutes at 250°F
- (c) 10 minutes at 325°F
- (d) 20 minutes at 250°F
- (e) 30 minutes at 150°F
- (f) 130 minutes at 75°F
- (g) repeat (a) through (f)
- (h) 18 hours at 75°F

The sequence of (a) through (h) was considered to be two cycles (see Figure 3) and was repeated on a five-day week basis. Thus a total 10 cycles per week were accumulated. The specimens were removed at intervals of 10 cycles exposure for their physical property characterization (new specimens each time).

c. Physical Testing Techniques

The physical testing of the materials investigated involved mechanical stress-strain, visual (photographics), and thermal gravimetric analysis. All mechanical testing was conducted at 74°F (23°C) and 50% RH. Materials under test are shown in Figure 4.

The foam materials were tested using 2 x 2 x 1 in. specimens according to ASTM Method D1621 at a crosshead speed of 0.1 in./min. The load deflection curves were recorded and are reported. The failed specimens were photographed.

For the thermoplastic materials, tensile testing was conducted using dog-bone type specimens in ASTM Method D638 at a crosshead rate of 0.05 in./min. The load compression curves were recorded and the specimens photographed.

The thermal gravimetric analysis was conducted in an American Instrument Company TGA apparatus using approximately 200 milligrams of unbroken specimen. Measurements were conducted in both air and helium, at a heating rate of approximately 5°F/min (3°C/min) from ambient to 1832°F (1000°C) (or until all weight was lost). A continuous recording of weight as a function of temperature was made and is reported.

Once the specimens were tested, they were not reused for any purpose.

2. THERMAL-RESISTANT FOAMS

Both coated and uncoated foams were evaluated on their ability to retain their mechanical properties upon exposure to Phase 1 and Phase 2 type environmental exposure.

The coated foams all consisted of the 10-pore per inch reticulated urethane foam coated with various high-temperature polymers. These polymers included nylon 11, polyimide, and polymer 380. The uncoated foams consisted of the 10-pore-per-inch reticulated urethane foams (as a reference), a polyester fiber foam, and polyimide foams.

Since it was most important for the fuel tank application, all foams investigated were quite porous and for the most part had large open cells.

Of significance to the coated urethane foams was the fact that their moduli were orders of magnitude greater than the original reticulated urethane. This is illustrated in Figure 5. In fact, while the original reticulated urethane foams would be considered flexible, the coated urethane would be considered rigid. The materials, therefore, would not be able to sustain any significant amount of deflection, followed by recovery. While not proven, doubt would have been placed as to whether the rigidized foam would perform any where near as well as the uncoated foam as a fire and explosion suppressant material.

The coated foams were all much more friable than the original urethane. The edges, therefore, could be readily broken causing the production of small pieces of material in the fuel tank.

The polyester fiber and the cured polyimide foams both exhibited a modulus near the range of the original 10-pore-per-inch reticulated urethane. It was felt that this was quite advantageous, since it would not preclude their utility as a fire and explosion suppressant material, while providing integrity at the elevated temperatures in the fuel environment.

a. Coated Foams

The concept behind the coated foams was to provide a thermally-resistant polymer to the surface of the urethane foam structure, which would partially protect and as a minimum maintain its structural integrity upon exposure to JP-5 fuel at elevated temperatures.

These foams were all prepared by the Scott Paper Company by undisclosed methods, which provided for approximately 100 weight percent of application (based on the urethane substrate) of each of the various high-temperature materials. The samples prepared consisted of urethane foams coated, respectively with a single dip of nylon 11, a double dip of nylon 11, American Cyanamid's 8692-192A polyimide, and 3M's polymer 380. It had originally been anticipated that nylon 66 would be investigated; however, Scott was unable to prepare a suitable sample. The results of the exposure of these coated foams under Phase 1 conditions are shown in Figures 6, 7, 8, and 9. As is readily shown, the mechanical performance of these foams degraded rapidly and for the most part integrity was sustained for no more than one week. The decomposition of these foams is also illustrated by the photographs in Figure 10.

Since the above mentioned coated foams did not perform well when subjected to Phase 1 exposure at 325°F, a determination of the limits up to which these foams may be useful was attempted. Therefore, Phase 2 type isothermal exposure at both 200 and 250°F was conducted. This modified exposure was conducted on the nylon 11 and polyimide-coated urethanes and the results are shown in Figures 11, 12, 13, and 14. A summary of these data showing mechanical performances as a function of both time and temperature are shown in Figures 15 and 16 for the nylon 11 and polyimide-coated urethane.

Since the nylon 11-coated urethane sustained the least degradation of any of the coated foams upon exposure to Phase 1 testing, it was investigated under Phase 2 exposure. The results of this Phase 2 exposure on the mechanical properties of this material are shown in Figure 17 and are summarized in Figure 18.

Some improvement in the performance of the coated foam was shown, as would have been expected, due to the less severe Phase 2 thermal exposure. It was desirable that the coated foam hold up for a period of at least 100 cycles. This condition, however, was not met and the coated foam could not qualify for the desired application.

In order to demonstrate the weight-loss characteristics as a function of temperature, all the coated foams were treated by thermogravimetric analysis, as described in section III.1.c. Each specimen was run individually in both air and helium atmospheres. The results of these analyses are shown in Figures 19 and 20.

What is demonstrated in these thermal analysis curves is the original degradation of the urethane at approximately 250°C followed by the decomposition of the various coatings at the higher temperatures relating to their thermal integrity. The improvement in retention of weight due to the addition of the various coatings is shown which is significant within the 5-hour time frame of the test.

b. Uncoated Foams

In this portion of the program we investigated various uncoated foams, which included the 10-pore-per-inch reticulated urethane, 3M's polyester fiber foam No. 1458-1, and various types of foams based on Monsanto Company's RI-7271 polyimide.

The uncoated reticulated urethane foam was examined primarily for reference and to establish the degree of thermal resistance available to it alone. The polyester fiber foam and the polyimide foam were reported to have excellent thermal resistance in the presence of aviation fuels and therefore were of interest.

The 10-pore-per-inch reticulated urethane foam was evaluated under regular and modified Phase 1 type isothermal conditions at 325°F, 200°F, and 250°F. The results of these exposures on the mechanical performance of the foam are shown in Figures 21, 22, and 23, and illustrated in Figures 24 and 25. For reference the color change of the JP-5 fuel is also shown. As the urethane foam was completely degraded after one week exposure to Phase 1 conditions at 325°F, it was necessary to evaluate this foam on a daily basis. It was shown that the foam was completely degraded (in terms of mechanical stability) within three days. The urethane foam was also evaluated under Phase 2 conditions; the results of this are shown in Figure 26.

The polyester fiber foam was supplied by the 3M Company as their Sample No. 1458-1. It consisted of a white, bulky, felt material. The fact that the material was a felt and rather bulky hindered our testing of the material in compression and resulted in a greater deviation of results. However, this fact did not detract from the performance of the fiber foam in the Phase 1 and Phase 2 type exposure.

The retention of the mechanical performance of the polyester fiber foam No. 1458-1 is well demonstrated in Figures 27 and 28, and illustrated in Figure 29 for periods of 10 weeks exposure at 325°F (Phase 1) and thermocyclic exposure from ambient to 325°F (Phase 2).

A polyimide foam was made available from Monsanto Company. It was designated as sample RI-7271, and consisted of a fine-celled foam (approximately 40 pores per inch) in densities of 1 and 6 pounds per cubic foot. While these foams were not directly suitable for the intended application (since the flow of fuel would be restricted), it was felt that the establishment of their mechanical performance as a function of Phase 1 and Phase 2 type exposures would be excellent and should be proven.

Both the 1- and 6-pound-per-cubic foot polyimide foams were exposed to Phase 1 and Phase 2 type conditions. The results are shown in Figure 30 and 31 and illustrated in Figure 29. In essence, little degradation of mechanical properties occurred over a period of ten weeks or 100 cycles.

Thermalgravimetric analysis data for both the polyester fiber and polyimide foams in air in an inert atmosphere are shown in Figures 32 and 33.

In an attempt to prepare a polyimide foam that would be more useful for the intended fuel tank application, polyimide foams were prepared using a salt ball extraction technique. This consisted of packing a container with spherical sodium chloride salt balls, filling the container with a weighed amount of polyimide foaming powder, foaming this powder under appropriate temperature-time cycles and dissolving away the salt. Foams so prepared are shown in Figure 34. The foams had large holes the size of the salt balls and, while suitable connecting holes did not result, the feasibility of preparing foams by this technique was determined. Most importantly, the modulus of this foam was close to that of the reticulated urethane foam (which was not the case in the polyimide-coated urethanes).

The large-celled polyimide foams were evaluated under Phase 1 and Phase 2 type exposure and the results of this exposure on the mechanical performance are shown in Figures 35 and 36. Again, the excellent retention of properties for this material was demonstrated.

3. THERMAL-RESISTANT THERMOPLASTICS

Thermoplastics, both filled and unfilled, were evaluated for their ability to retain their mechanical properties upon exposure to Phase 1 and Phase 2 type environments.

The filler for all the thermoplastics was glass at a loading of 15% or 30%. The filled thermoplastics consisted of two different glass-filled nylons, and glass-filled polyester, polychlorotri-fluoroethylene, polyvinylidene chloride, polycarbonate, and

polytetrafluoroethylene. The glass was all in a short fiber form. The unfilled thermoplastics consisted of polyimide, nylon 66, polymer 360, polyphenylene oxide, and polysulfone. It was anticipated that some of these materials, such as polycarbonate, would not be effective in this particular application, but these were briefly screened to demonstrate the problem.

The purpose in investigating these thermoplastics was for their anticipated use in injection molding of hollow, perforated spheres. About midway through the program, however, the Air Force indicated a lack of interest in the hollow, perforated spheres for the fire- and explosion suppressant applications. Therefore, less emphasis was placed on these materials. Importantly, however, some of these materials were investigated, i.e., polyimides, polymer 360, and the nylons, to establish their utility, prior to their inclusion as a coating for the foam-type materials discussed in section III.2. Because of the aborted interest, the filled and unfilled thermoplastics were exposed only to Phase 1 type conditions.

a. Filled Thermoplastics

The reason for investigating the filled thermoplastics was their reported stability at elevated temperatures (+300°F) in aviation fuel environments. However, it was not anticipated that the glass filler would contribute any improved thermal or fuel resistance to the polymer. But the contribution of the glass to the modulus of the material was expected and proven to be useful in retaining the geometric integrity of the various specimens in the Phase 1 type exposure. It is most important to consider that the testing of the thermoplastics was done at 74°F (23°C) and not at the temperature of exposure.

The specimens were all obtained from the manufacturer, for the most part in the form of molded ASTM-D-638 dog-bone tensile specimens. The specific materials that were investigated included: LNP Corporation glass-filled (30%) nylon 66, No. RF-1006; DuPont glass-filled (30%) nylon 66, Zytel 7010-33; LNP Corporation glass-filled (30%) polyester, WF-1006; 3M Company glass-filled (15%) polychlorotrifluoroethylene, KF-6066; and Pennsalt Company glass-filled (15%) polyvinylidene chloride, Kynar 315MRN; glass-filled polycarbonate; and glass-filled polytetrafluoroethylene.

The mechanical performance of the first five materials, as a function of Phase 1-type exposure at one week increments, is shown in Figures 37 thru 41. The tensile strengths, modulus of elasticity, and percent elongation as a function of exposure time for the nylons and polyester are shown in Table I. Photographs of the failed specimens following the various exposures

are shown in Figures 42 and 43. This includes the glass-filled polycarbonate and Teflon, which could not be tested after exposure.

As shown in Figures 37 and 38 the glass-filled nylons exhibited excellent retention of properties as a function of exposure time. The glass-filled polyester exhibited considerable decrease in tensile strength in just one week exposure, and the polyvinylidene chloride, the polychlorotrifluoroethylene, and the polycarbonate all lost their initial geometry in the exposure so that no more mechanical performance tests could be conducted.

b. Unfilled Thermoplastics

The unfilled thermoplastics were investigated both for their potential interest by themselves and as coatings for the reticulated urethane foams. Since coating of the urethane foam with various materials was not exactly straight-forward, and since the materials were not off-the-shelf, it was advisable to first qualify the coating material. Specifically, testing of the polyimides and the polymer 360 were conducted for this purpose.

The unfilled thermoplastic materials investigated consisted of American Cyanamid Company's polyimide 8692-192A (XPI-MC154); Union Carbide Company's polysulfone P; 3M Company's polymer 360; General Electric Company's polyphenylene oxide PPO; and a commercial grade (Cadillac Plastics) nylon 66 sheeting. The results of the exposure of two of these materials to Phase 1 conditions on mechanical performance are shown in Figures 44 thru 46. As demonstrated, both the polyimide and the polymer 360 exhibited excellent retention of mechanical properties as a function of exposure time under Phase 1 conditions.

The lack of retention of physical geometry of the polysulfone, polyphenylene oxide and nylon 66 are shown in the photographs (Figure 46).

APPENDIX

Table
Figures

Preceding Page Blank

TABLE I
PHYSICAL PROPERTIES OF THERMOPLASTIC MATERIALS AT 74°F BEFORE
AND AFTER IMMERSION IN JP-5 FUEL AT 325°F.

Specimen Description	Immersion Time (Days)	Tensile Strength (psi x 10 ³)	Modulus Of Elasticity (psi x 10 ⁶)	Elongation (%)
LNP Corp. glass filled Nylon No. RP-1006	0	22.3	1.38	2.1
"	7	22.4	1.21	2.9
"	14	21.6	1.35	2.4
"	21	20.6	1.25	2.3
"	28	19.4	1.22	2.0
E.I. Dupont glass filled Nylon Zytel 7010-33	0	18.8	1.03	4.1
"	7	24.9	1.49	2.5
"	14	22.3	1.38	2.2
"	21	19.8	1.67	1.5
"	28	17.2	1.45	1.4
LNP Corp. glass filled Polyester No. WF-1006	0	14.3	1.38	1.1
"	7	3.5	1.23	0.5
"	14	3.4	1.17	0.4
"	21	3.3	1.41	0.4
"	28	3.1	1.29	0.5
American Cyanamid Polyimide XPI-MC-154	0	13.5	0.51	3.9
"	7	6.8	0.50	1.5

Preceding Page Blank

Reproduced From
Best Available Copy

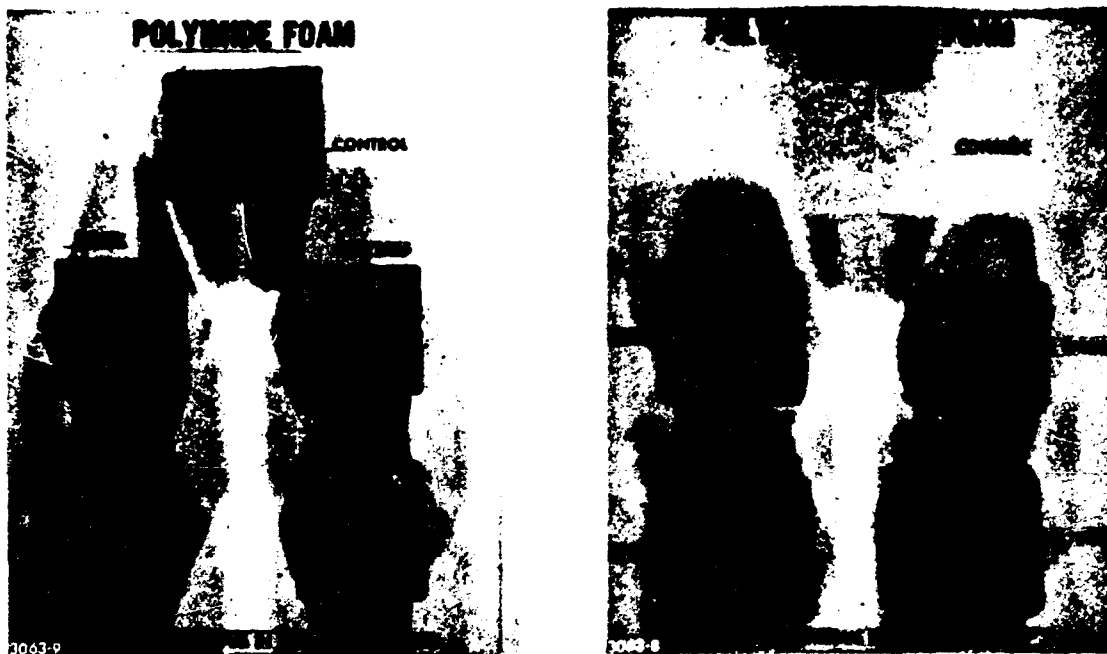


Figure 1. Foam Type Structures which Showed Promise for Use at 325°F in JP-5 Fuel for Extended Periods of Time.

Preceding Page Blank



(a) General View



(b) Specimen Container

Figure 2. Environmental Baths Used to Expose Candidate Materials in JP-5 Fuel at Temperatures up to 325°F.

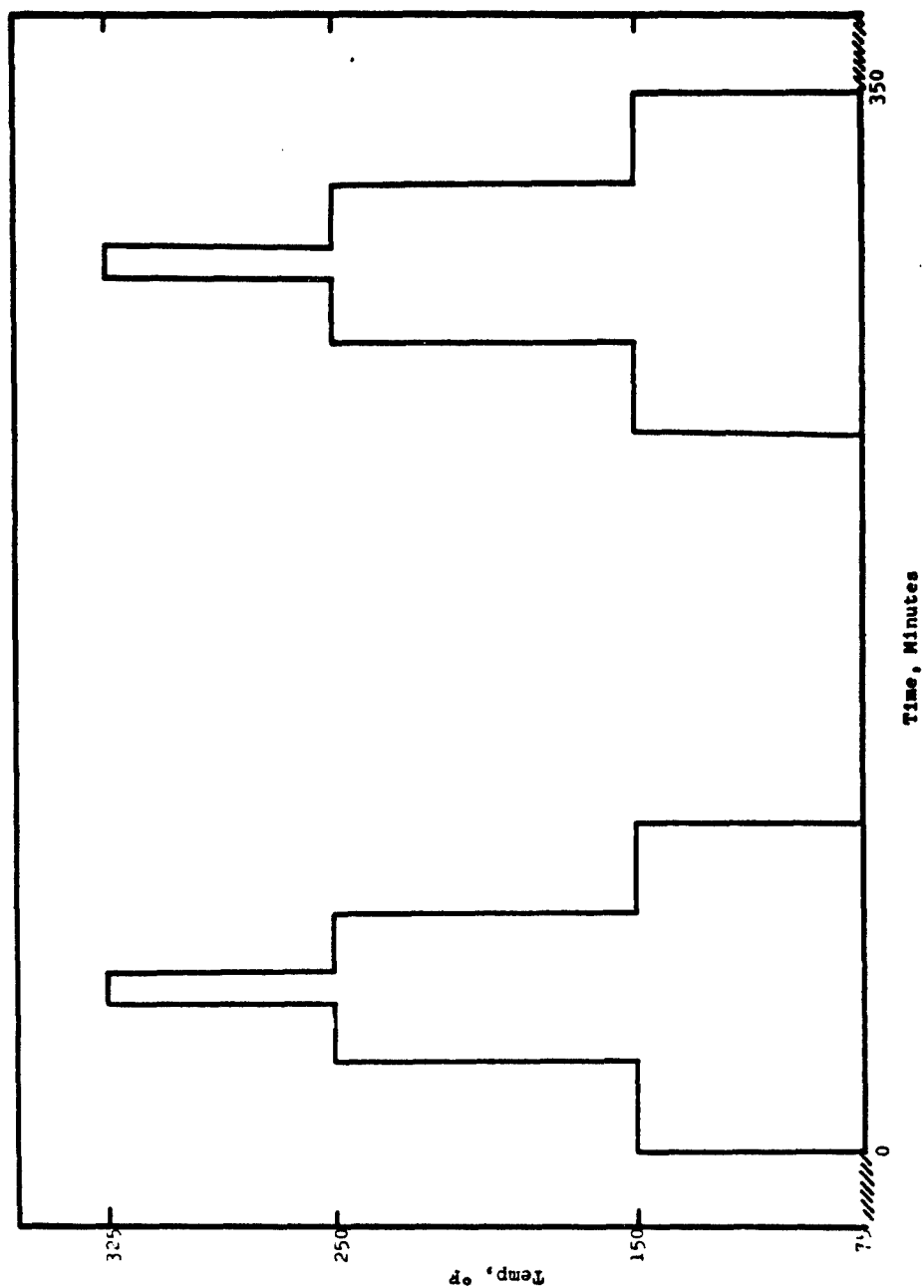
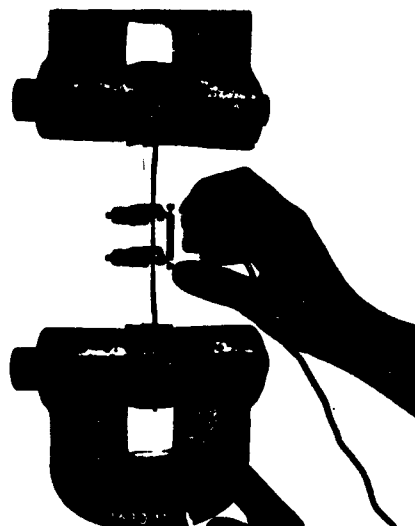


Figure 3. Time-temperature Sequence for Phase 2 Type Thermocycling.



(a) Compression Test



(b) Tensile Test

Figure 4. Test Configurations and Arrangements Used To Characterize Candidate Foam and Thermoplastic Materials.



POLYESTER
FIBER FOAM

URETHANE

POLYIMIDE

COATED
URETHANE

Figure 5. Photograph Illustrating the Modulus Characteristics of the Coated and Uncoated Foams.

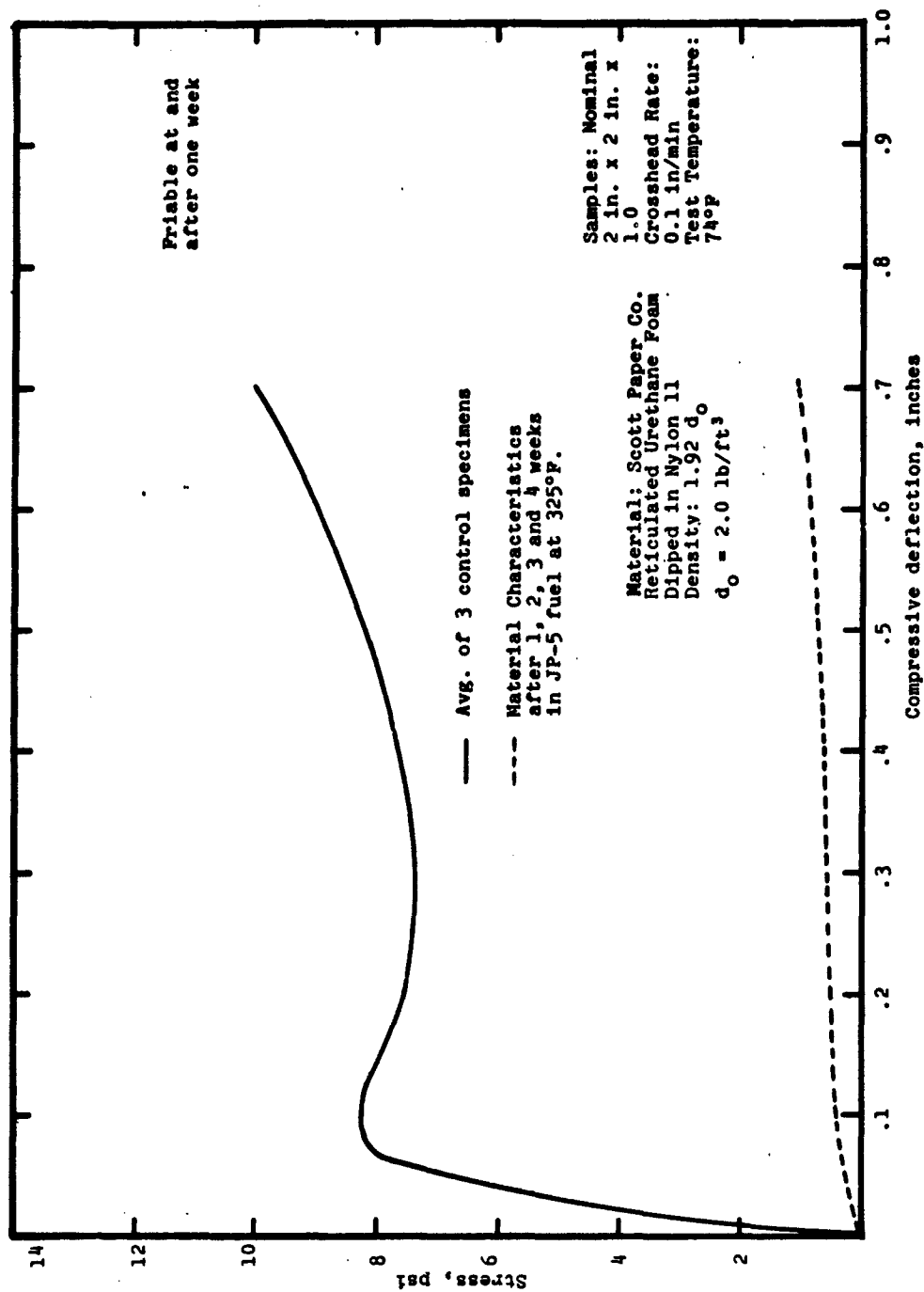


Figure 6. Stress vs Compression for Nylon 11 Single Coated Urethane Foam Following Phase 1 Exposure for Four Weeks.

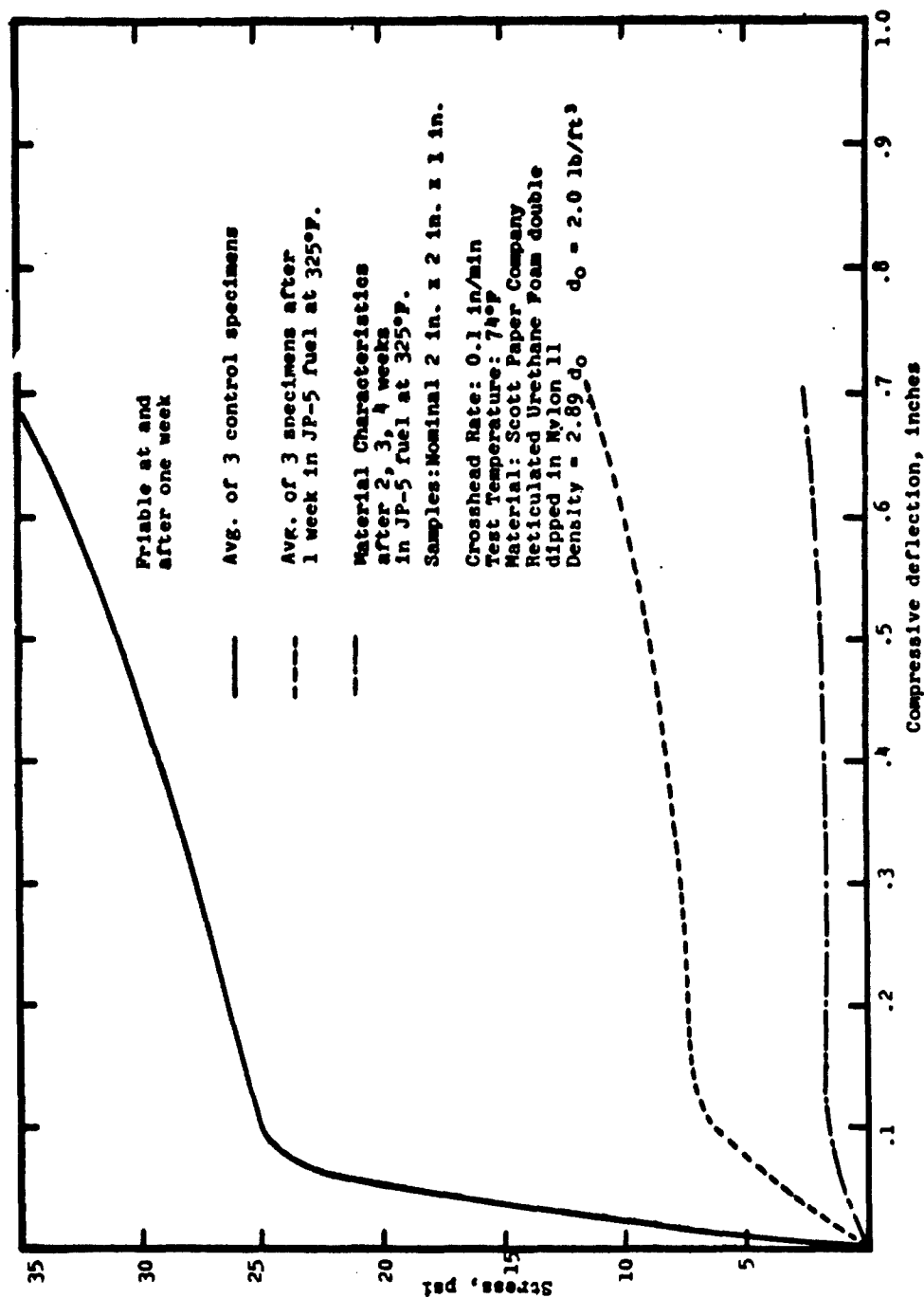


Figure 7. Stress vs Compression for Nylon 11 Double Coated Urethane Foam Following Phase 1 Exposure for Four Weeks.

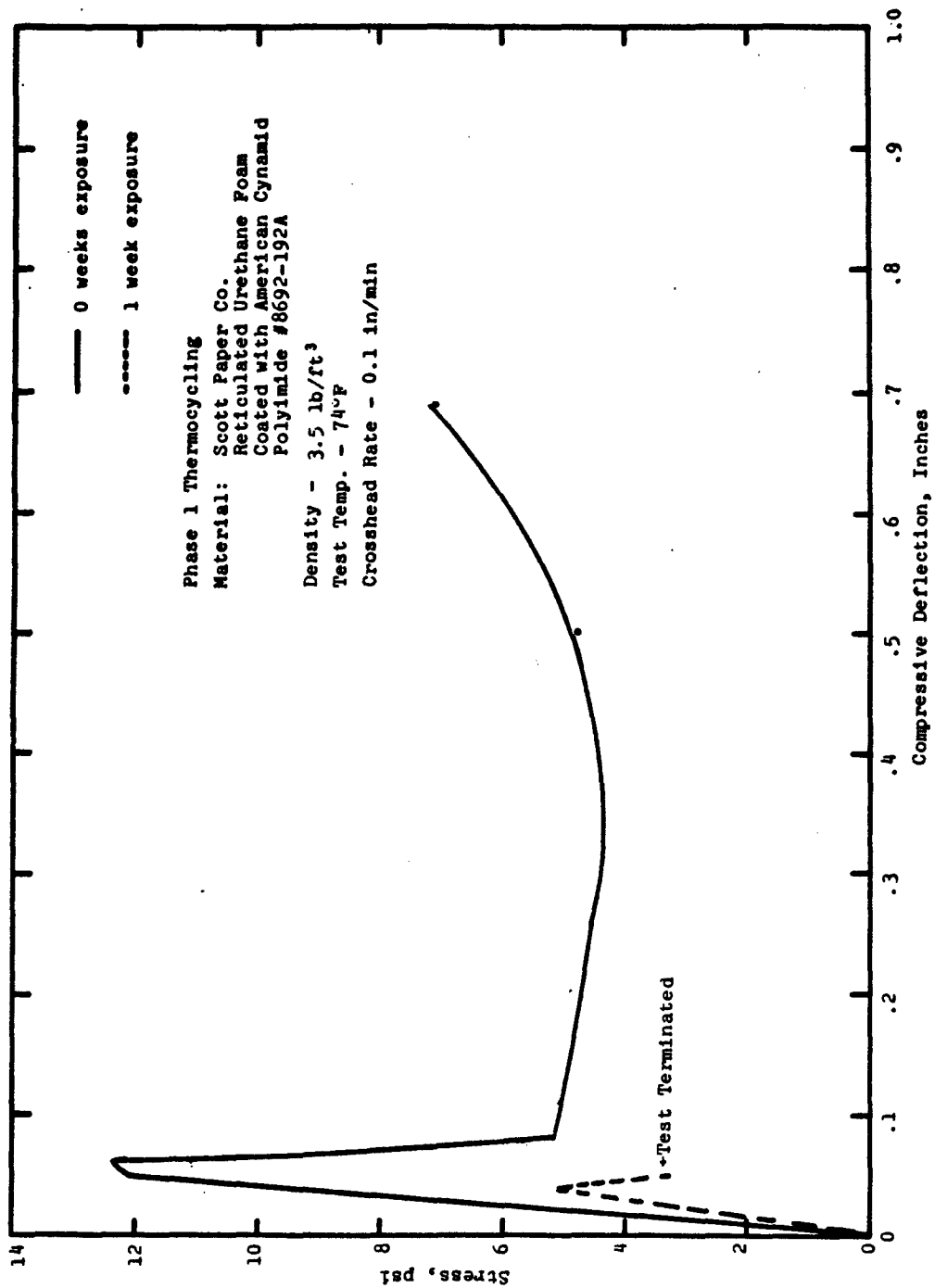


Figure 8. Stress vs Compression for Reticulated Urethane Foam Coated with Polyimide Following Phase 1 Exposure for One Week.

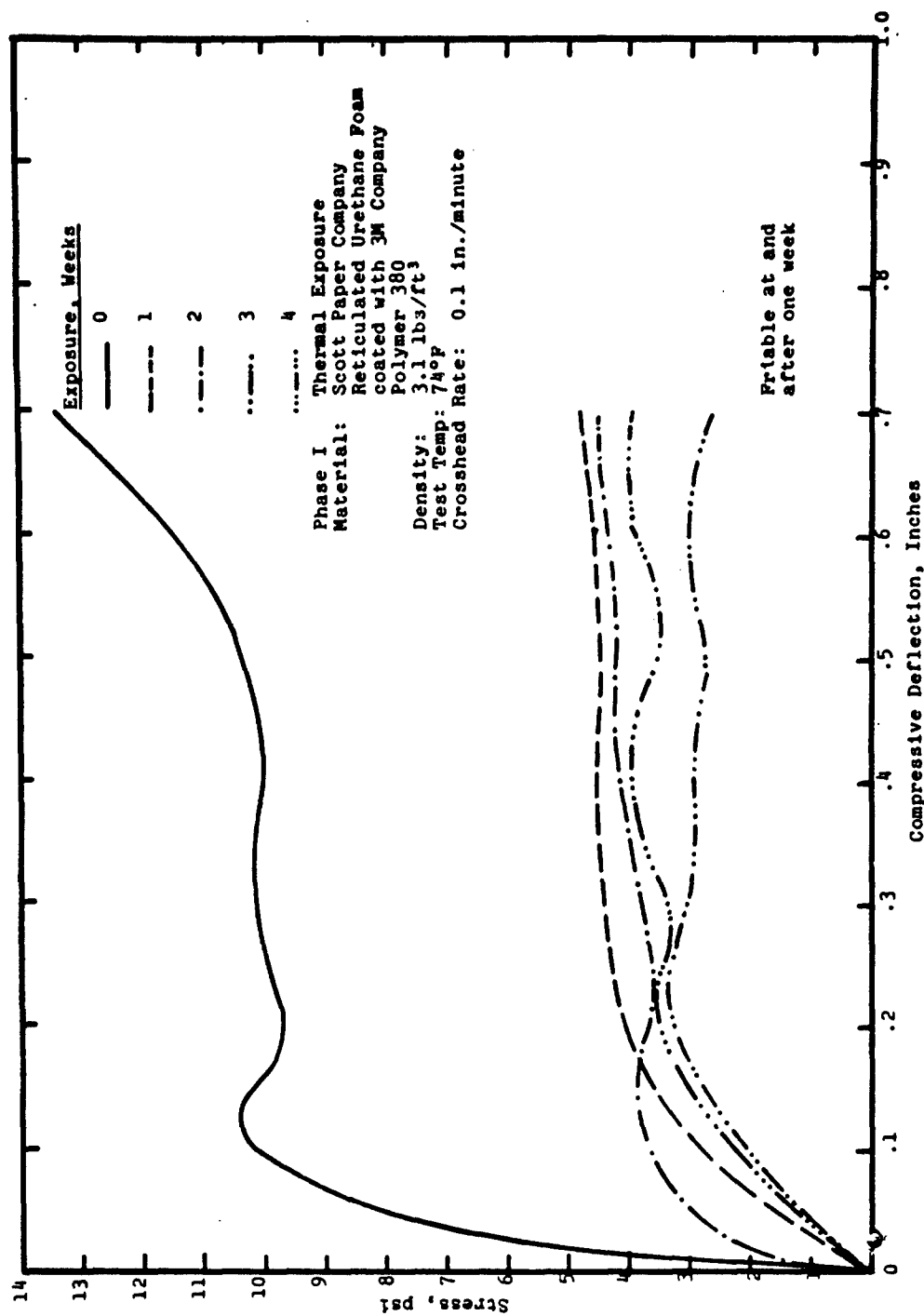


Figure 9. Stress vs Compression for Reticulated Urethane Foam Coated with Polymer 380 Following Phase 1 Exposure for Four Weeks.

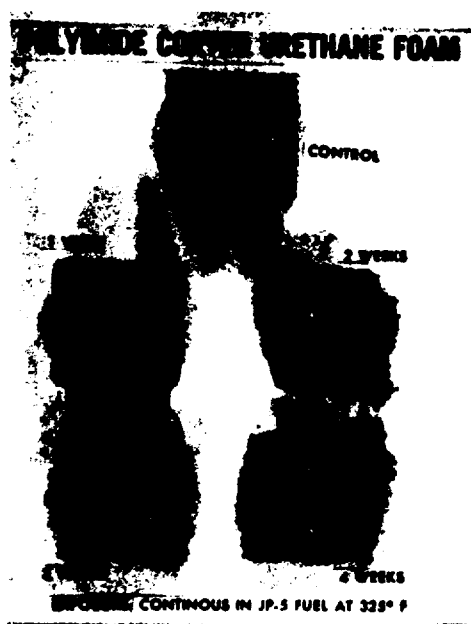
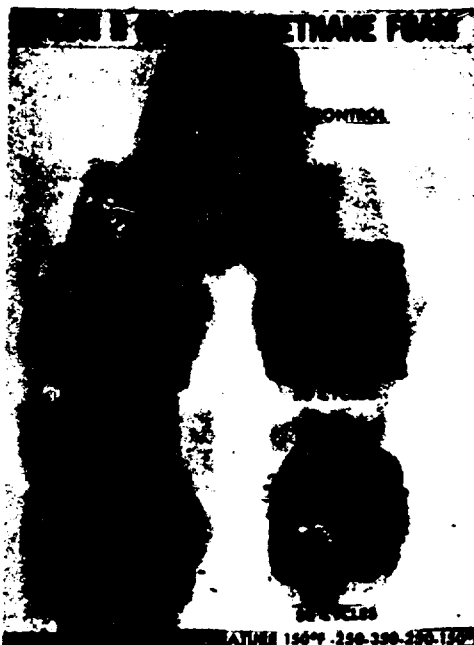


Figure 10. Photographs of Coated Urethane Foams Following Exposure to JP-5 Fuel at Elevated Temperatures.

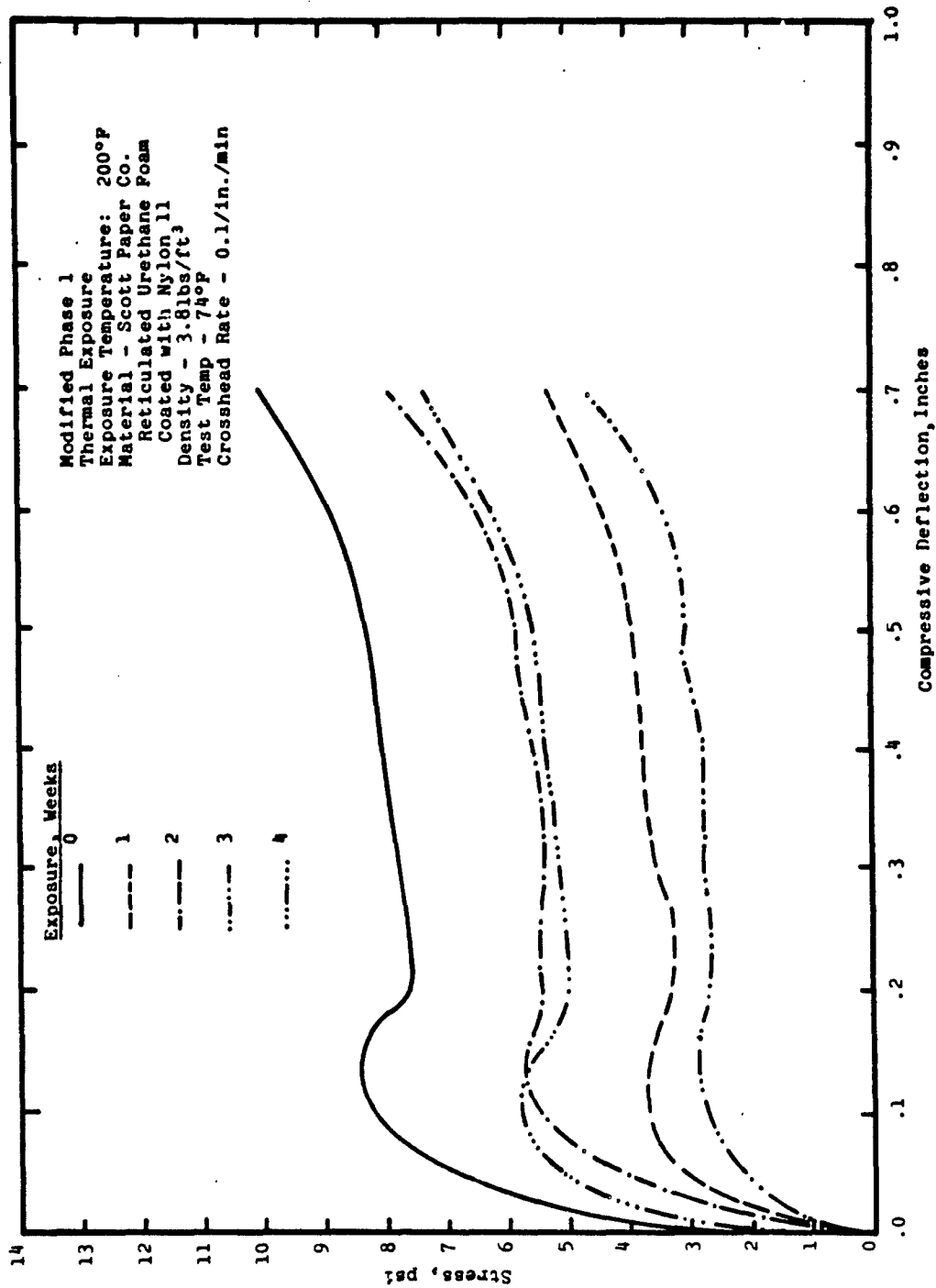


Figure 11. Stress vs Compression for Reticulated Urethane Foam Coated with Nylon 11 Following Exposure at 200°F in JP-5 Fuel for Four Weeks.

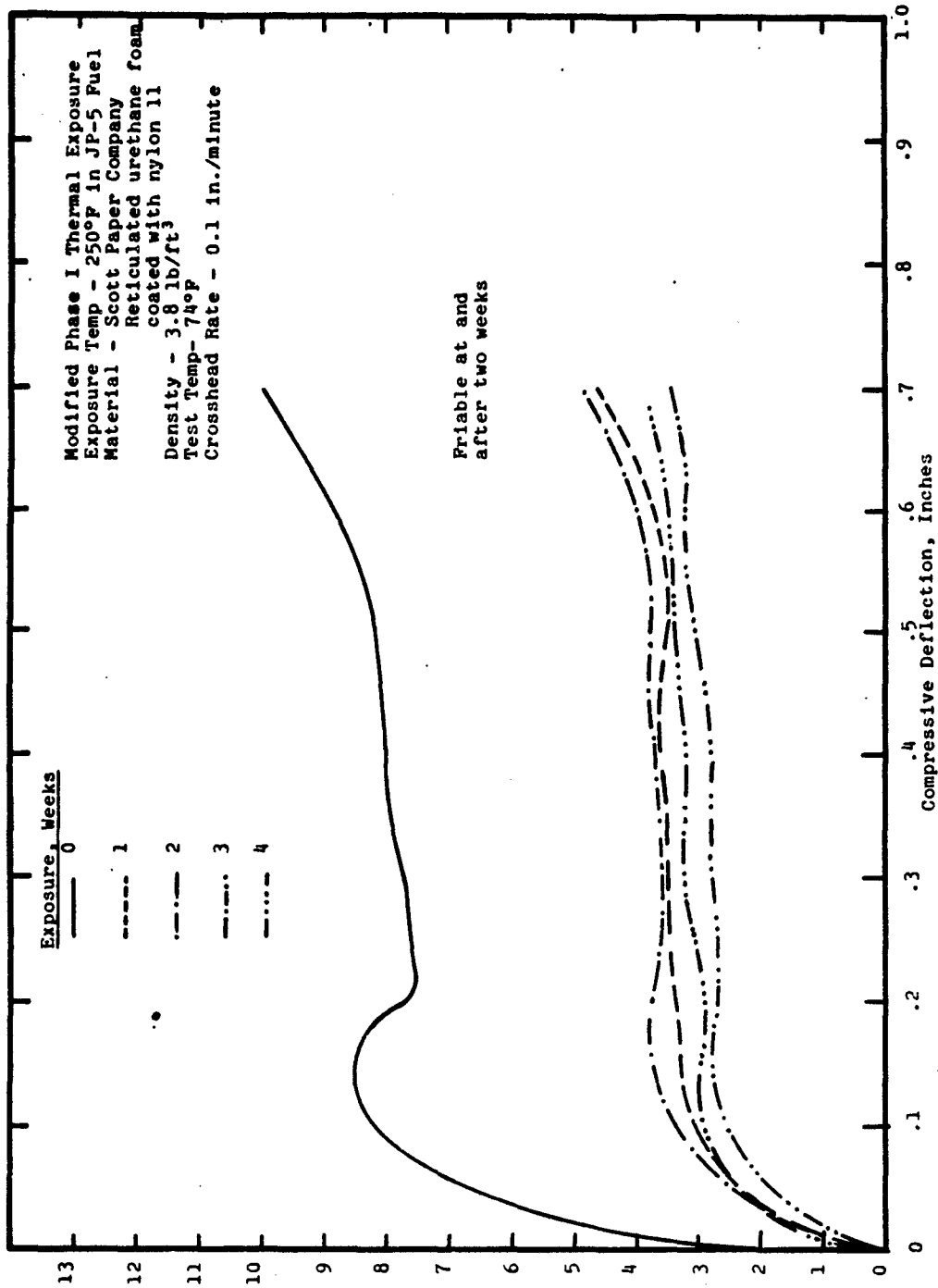


Figure 12. Stress vs Compression for Reticulated Urethane Foam Coated with Nylon 11 Following Exposure at 250°F in JP-5 Fuel for Four Weeks.

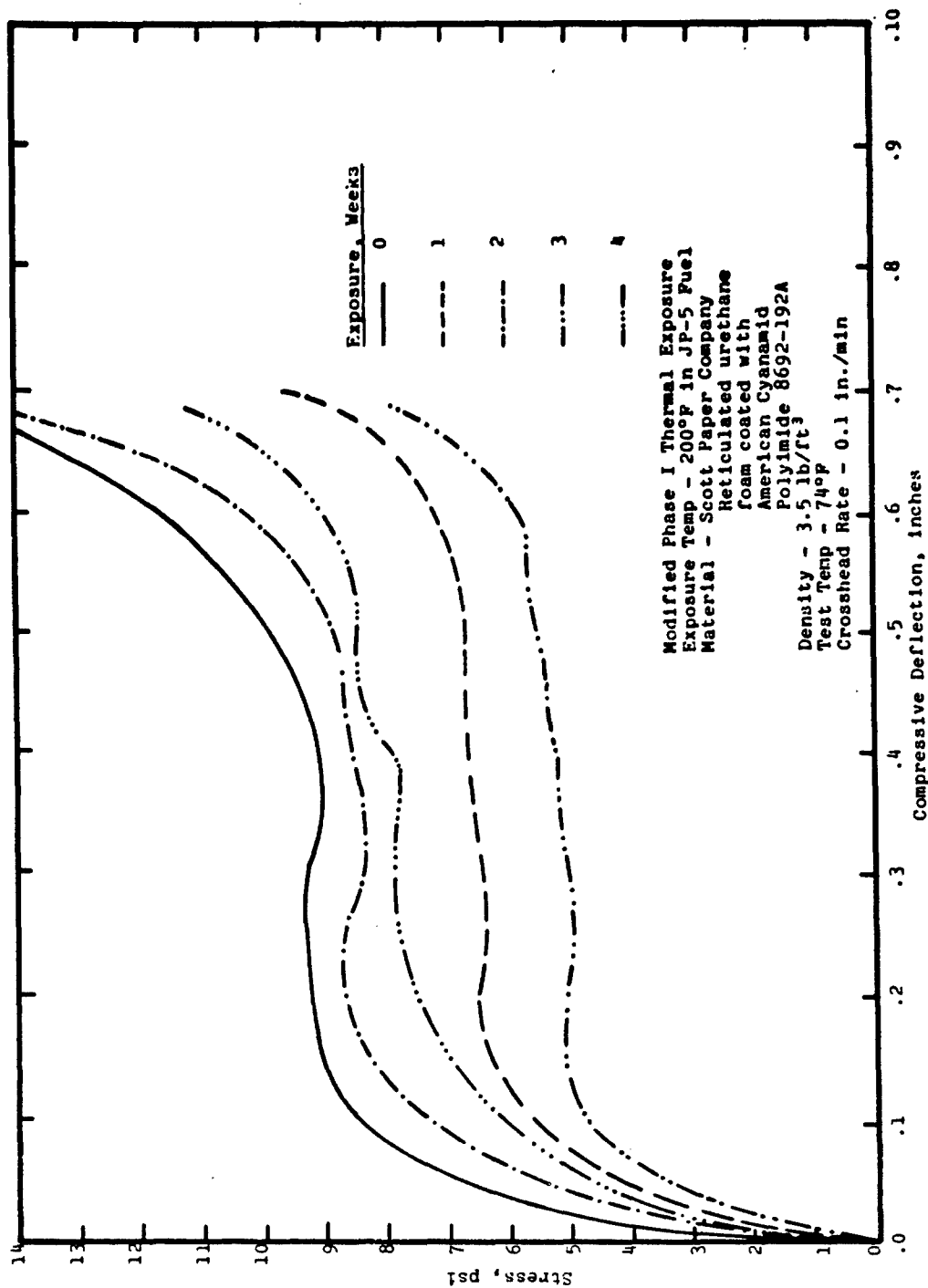


Figure 13. Stress vs Compression for Reticulated Urethane Foam Coated with Polyimide Following Exposure at 200°F in JP-5 Fuel for Four Weeks.

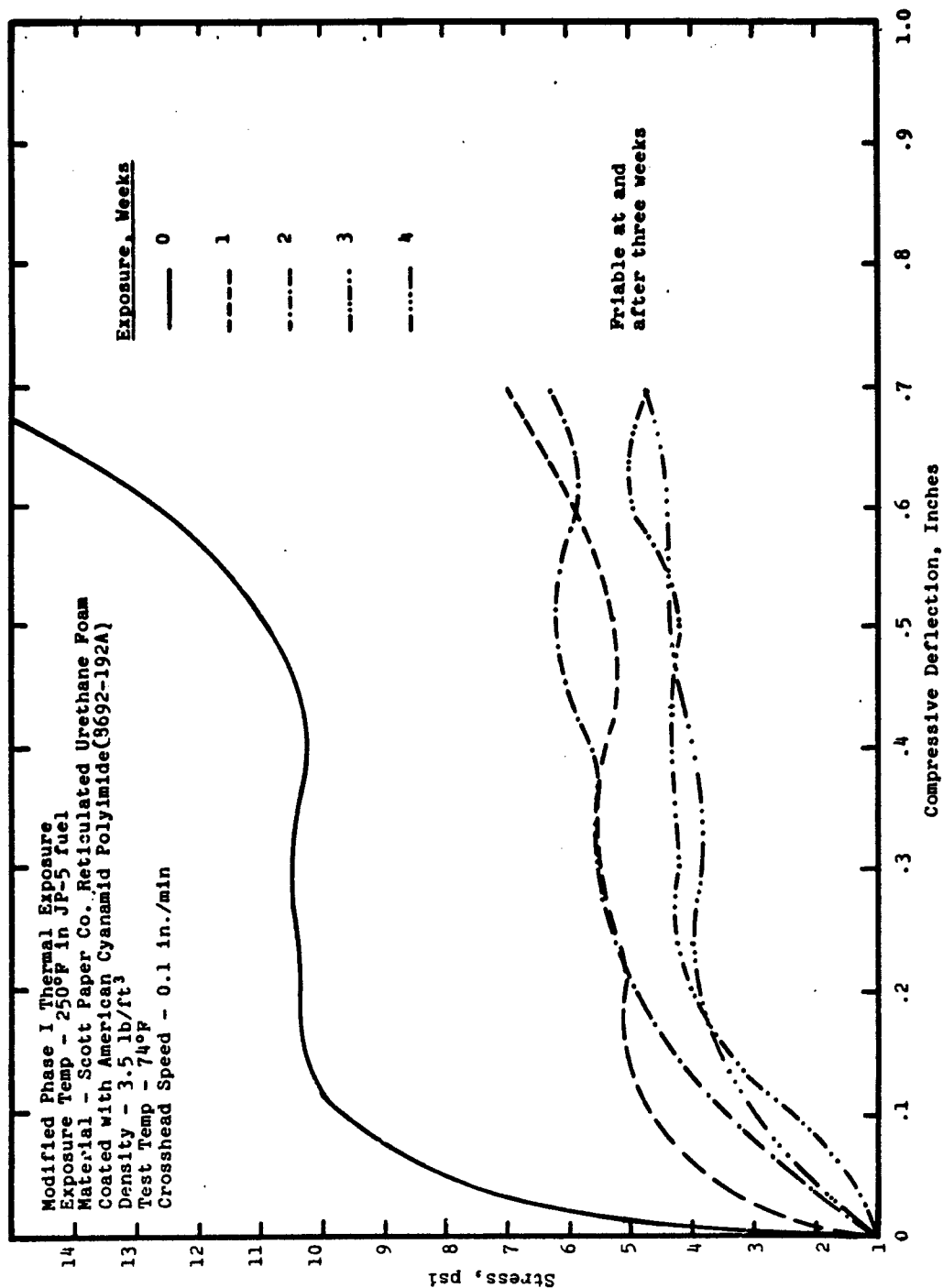


Figure 14. Stress vs Compression for Reticulated Urethane Foam Coated with Polyimide Following Exposure at 250°F in JP-5 Fuel for Four Weeks.

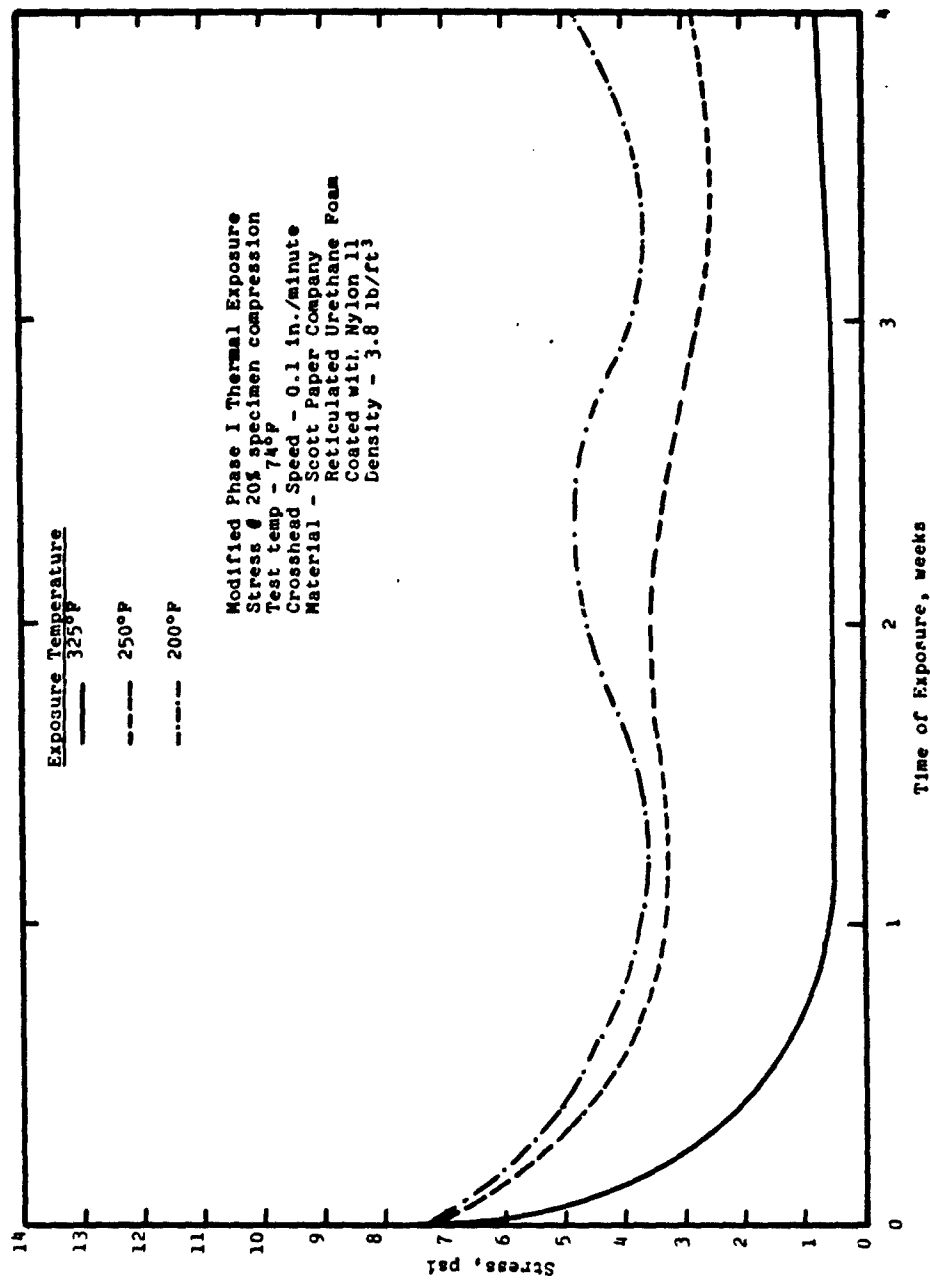


Figure 15. Stress vs Time of Exposure and Exposure Temperature for Reticulated Urethane Foam Coated with Nylon 11.

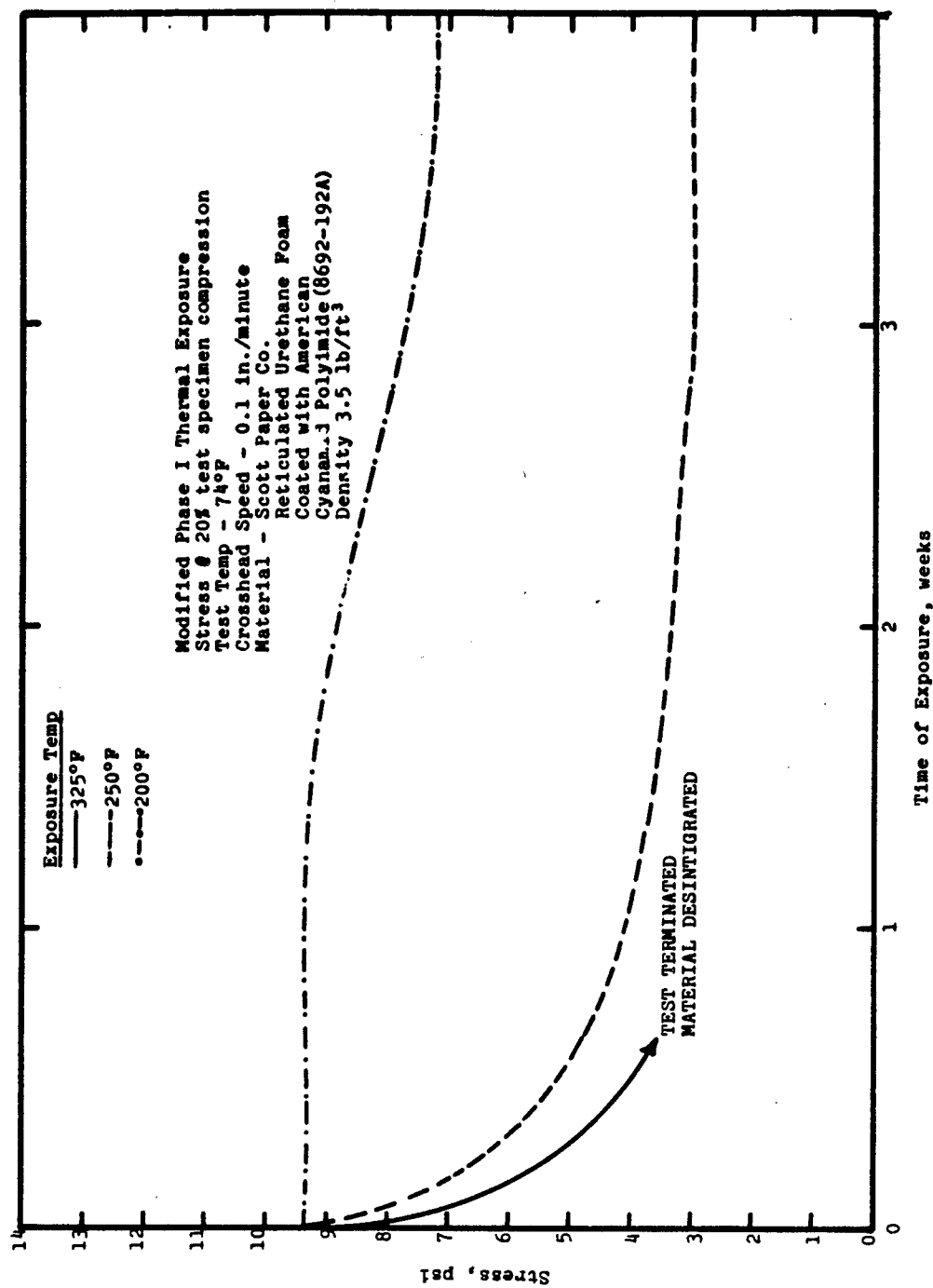


Figure 16. Stress vs Time of Exposure and Exposure Temperature for Reticulated Urethane Foam Coated with Polyimide.

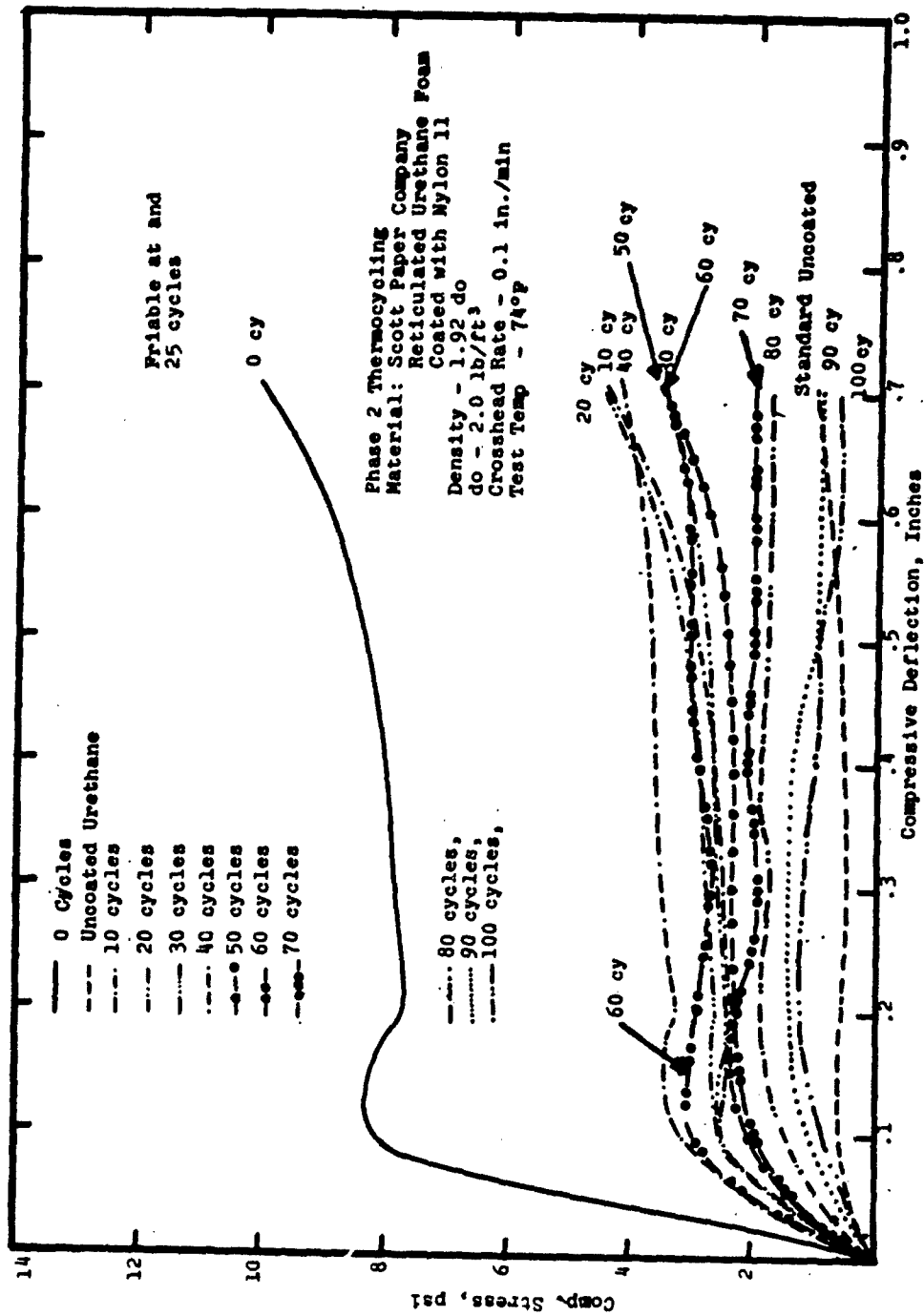


Figure 17. Stress vs Compression for Nylon 11-coated Urethane Foam Following Phase 2 Thermocycling for 100 Cycles (ten weeks).

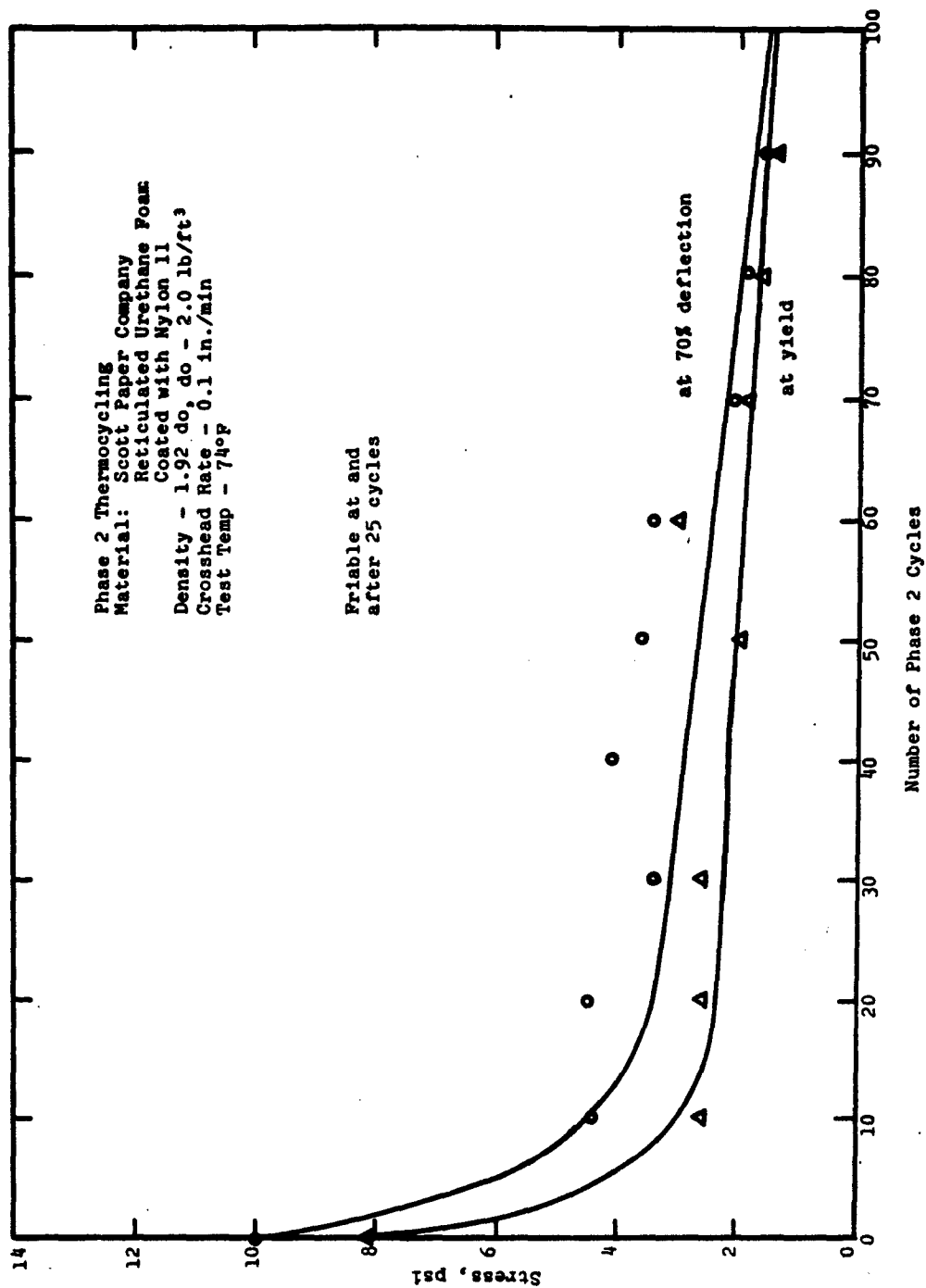


Figure 18. Compressive Stress as a Function of Exposure Time to Phase 2 Thermocycling for Nylon 11-coated Urethane Foam for 100 cycles (ten weeks).

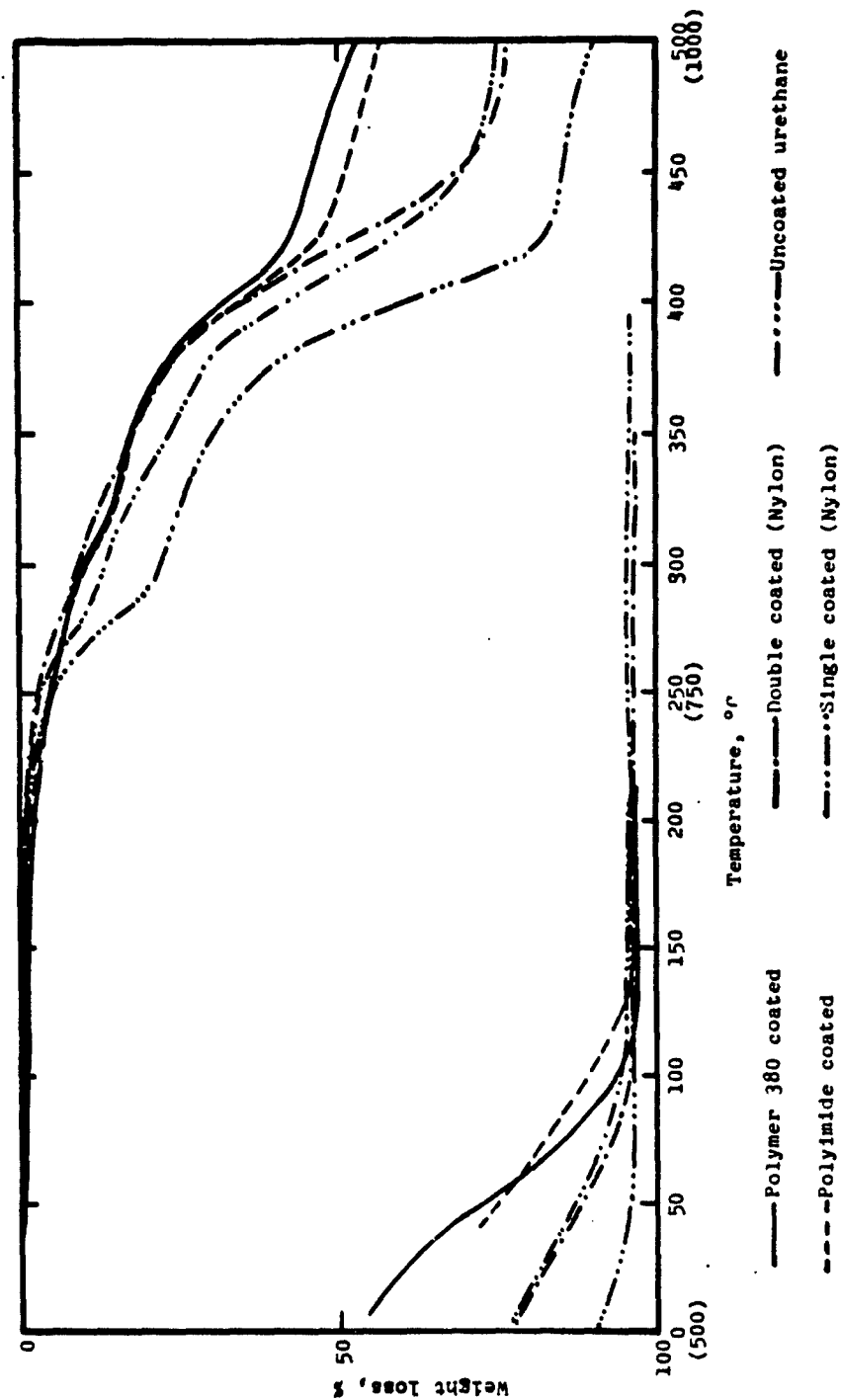


Figure 19. Thermal Gravimetric Analysis of the Uncoated and Coated Urethane Foams in Air.

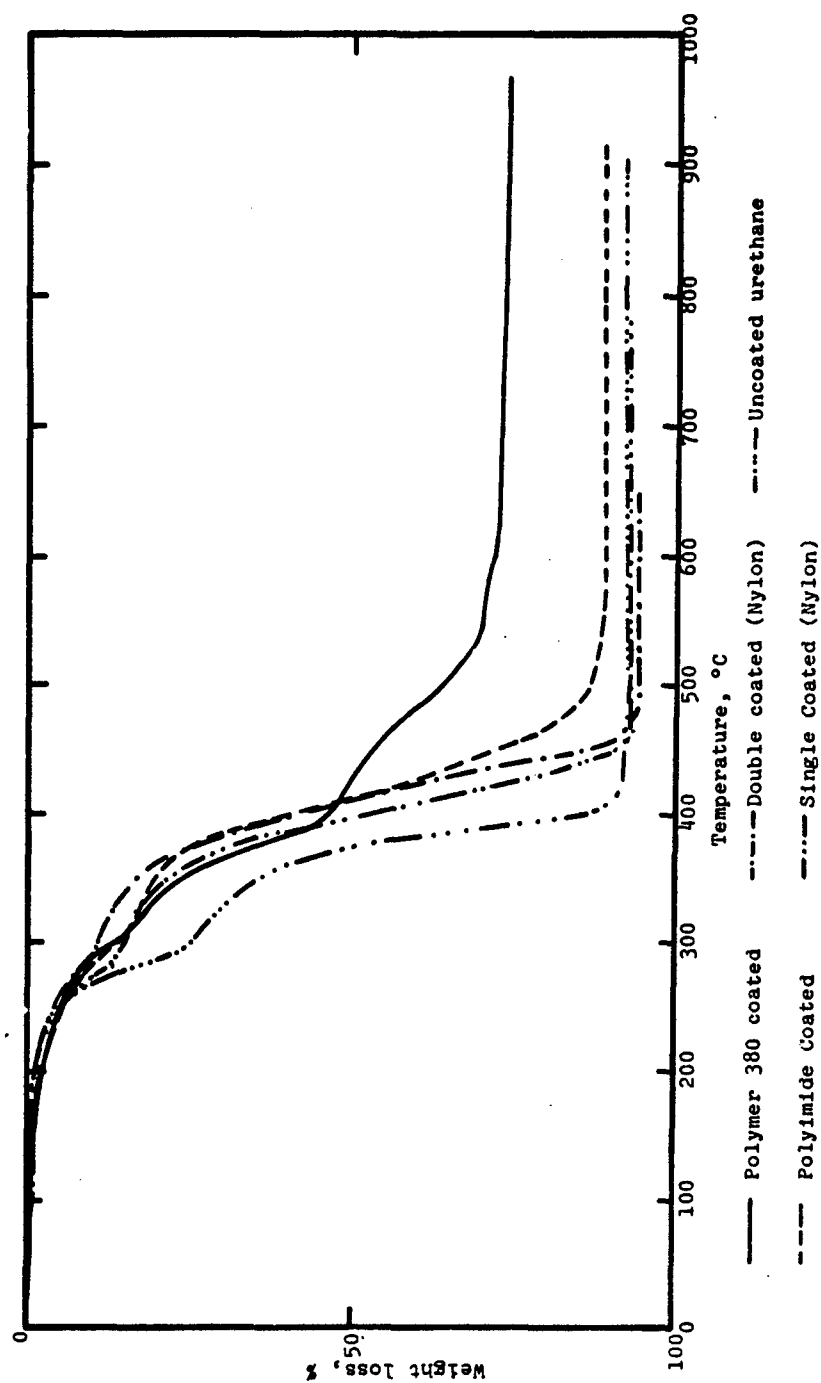


Figure 20. Thermal Gravimetric Analysis of the Urethane and Coated Urethane Foams in an Inert Atmosphere.

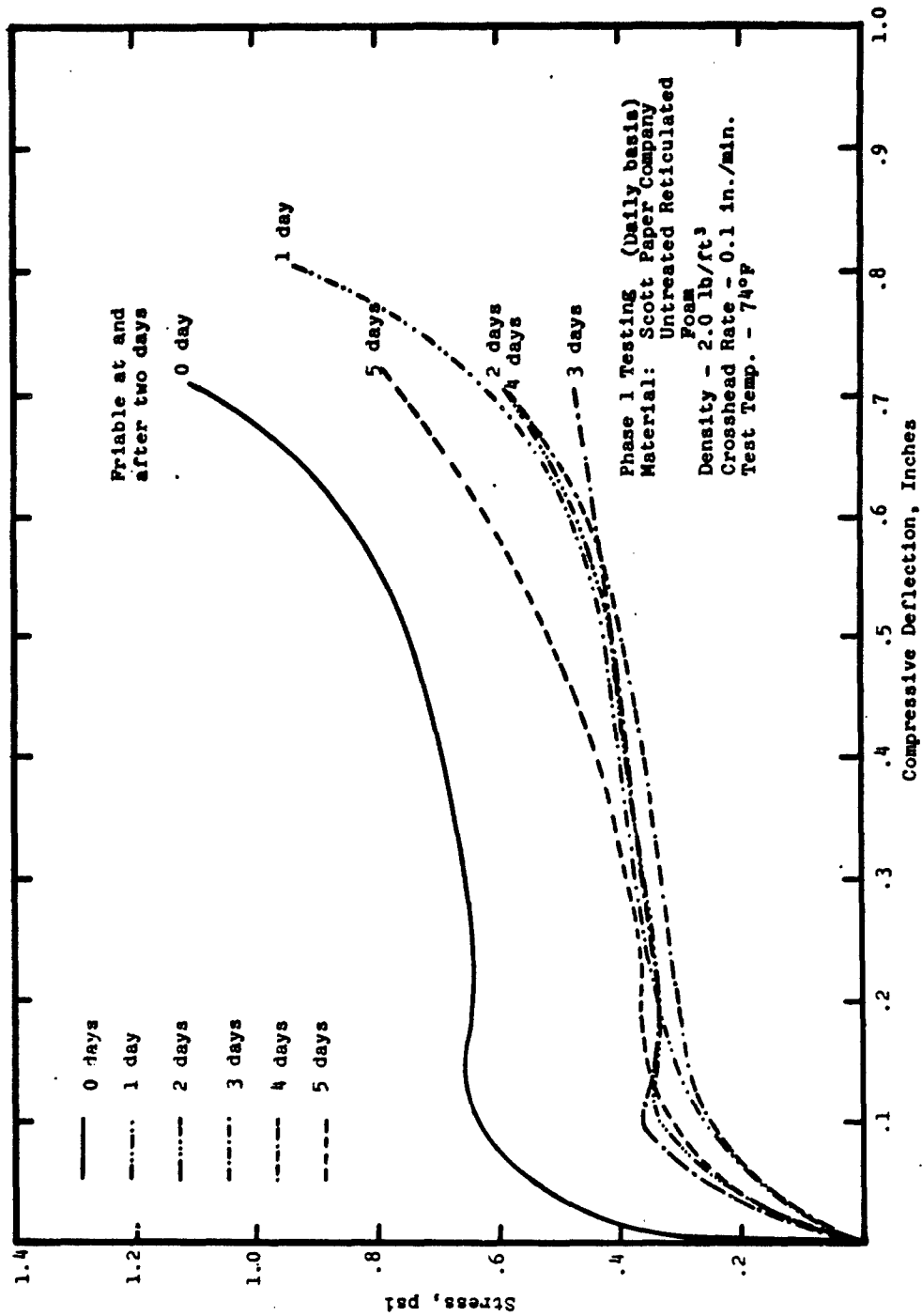


Figure 21. Stress vs Compression for Untreated Reticulated Urethane Foam Following Exposure at 325°F in JP-5 Fuel for Five Days.

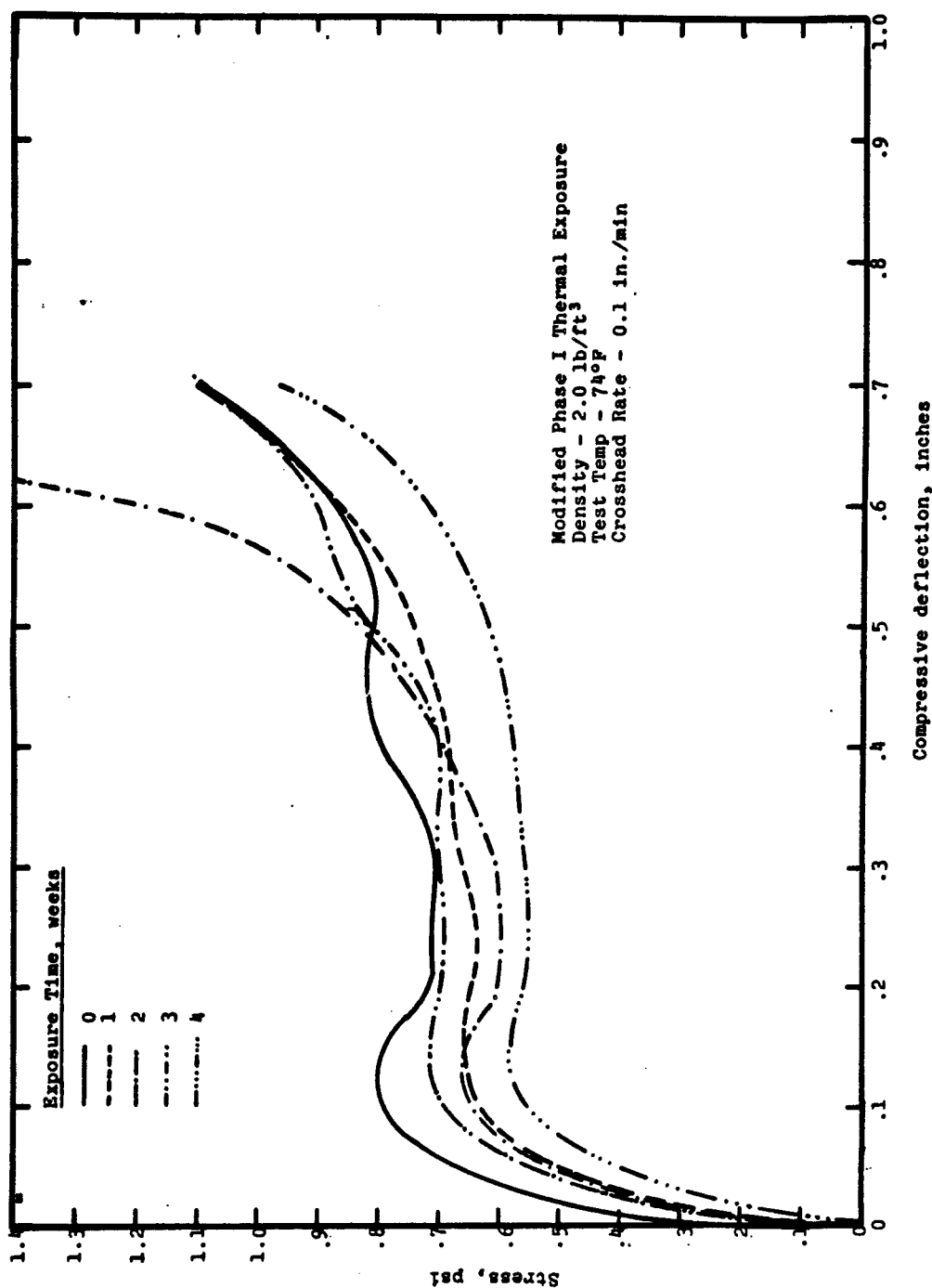


Figure 22. Stress vs Compression for Uncoated Reticulated Foam Following Exposure at 200°F in JP-5 Fuel for Four Weeks.

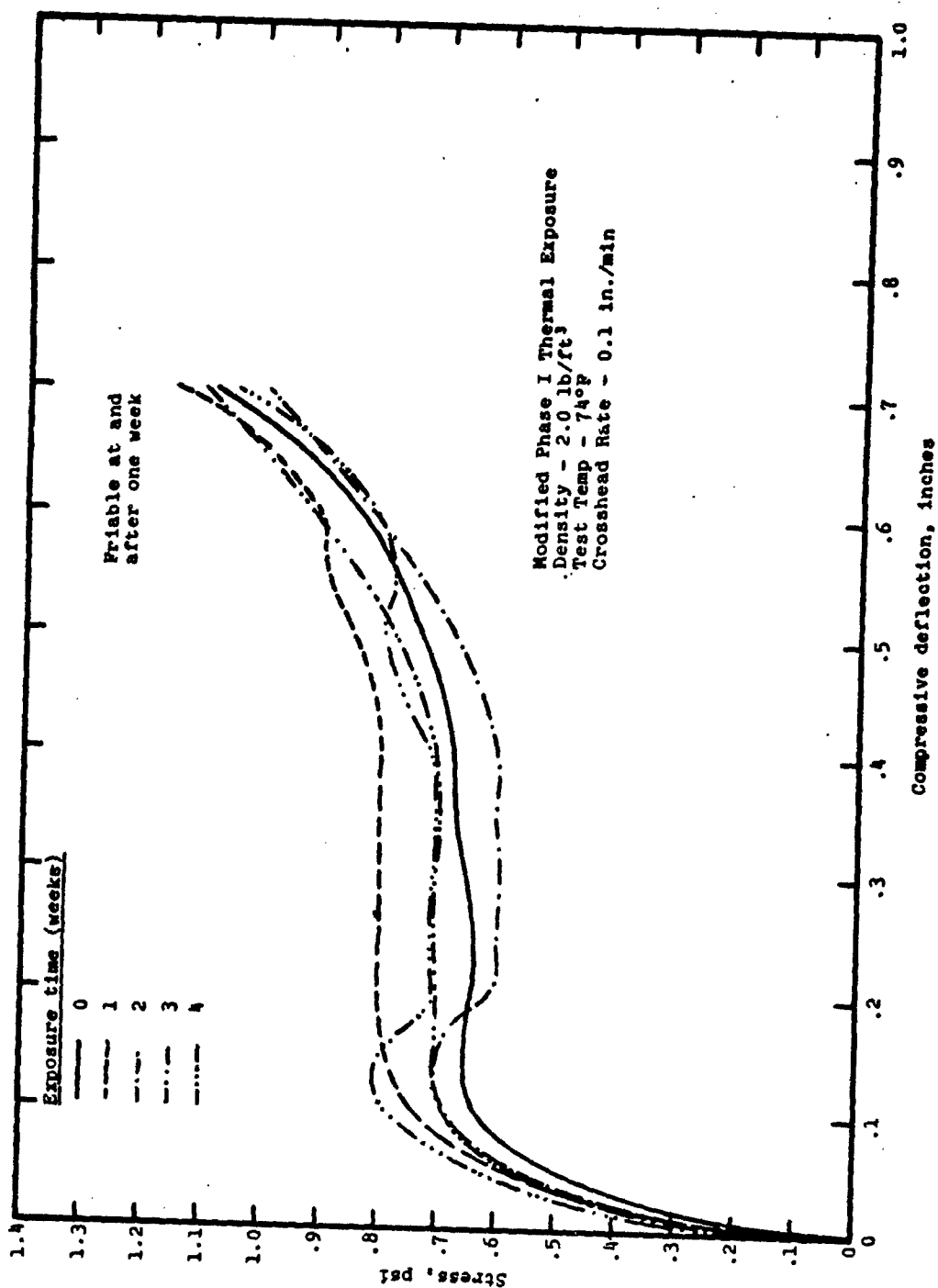


Figure 23. Stress vs Compression for Uncoated Reticulated Urethane Foam Following Exposure at 250°F in JP-5 Fuel for Four Weeks.

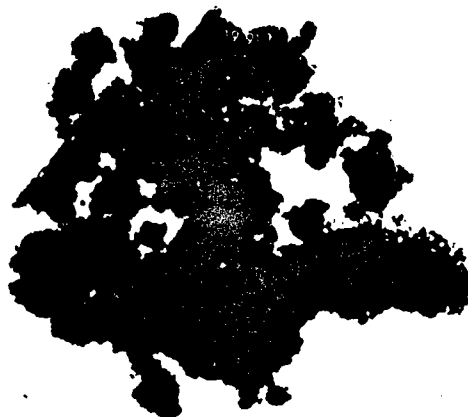


Figure 24. Reticulated Urethane Foam Following 1 Week
Immersion in JP-5 Fuel at 325°F (Phase 1 Exposure).



(Reference Color Change of JP-5 due to 325°F Exposure)

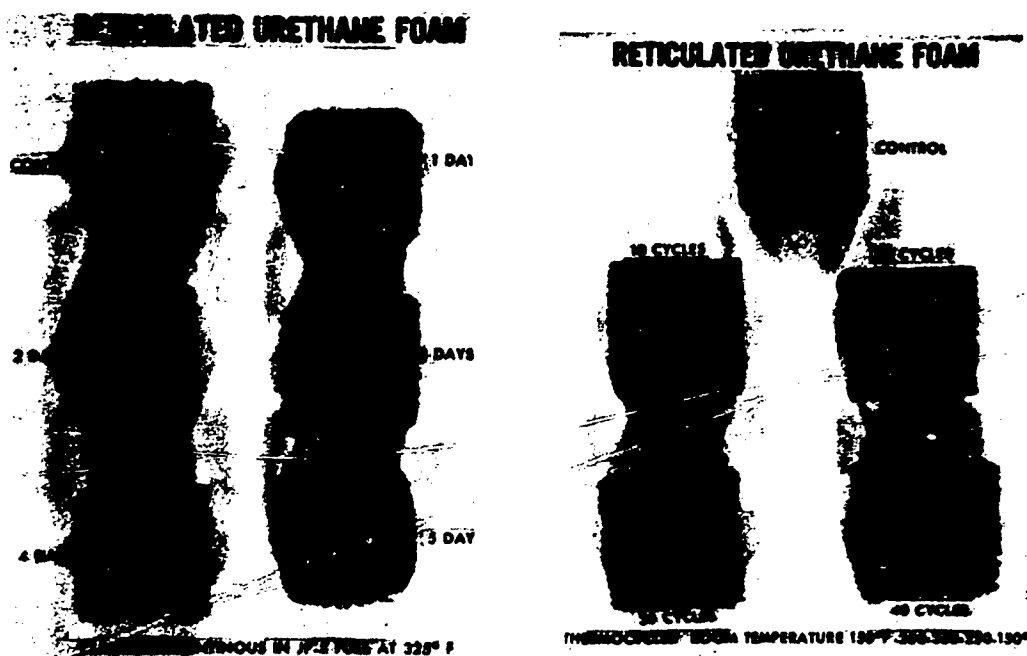


Figure 25. Photographs of Uncoated Urethane Foams Following Exposure to JP-5 Fuel at Elevated Temperatures.

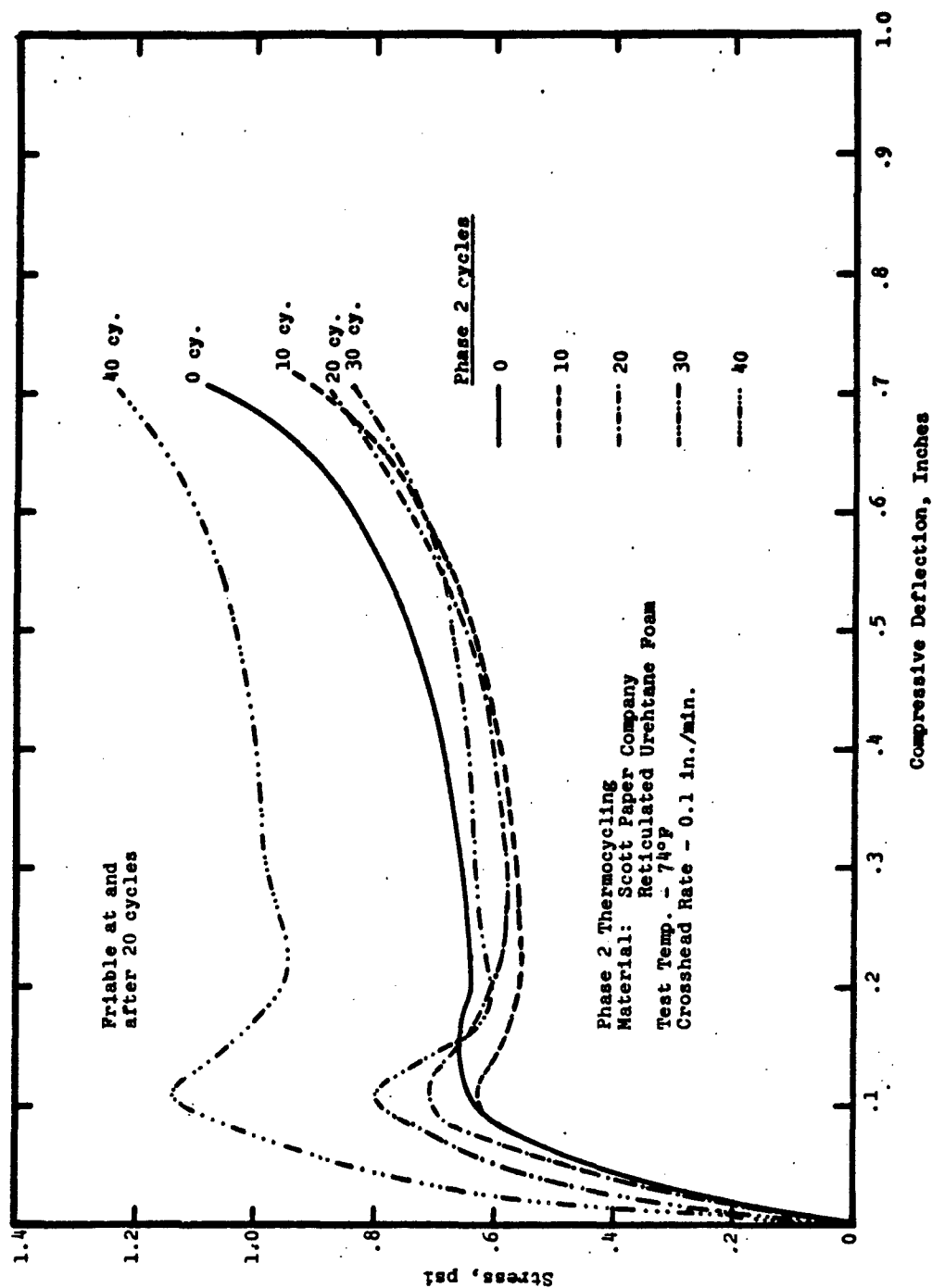


Figure 26. Stress vs Compression for Uncoated Reticulated Urethane Foam Following Phase 2 Thermocycling for 40 Cycles.

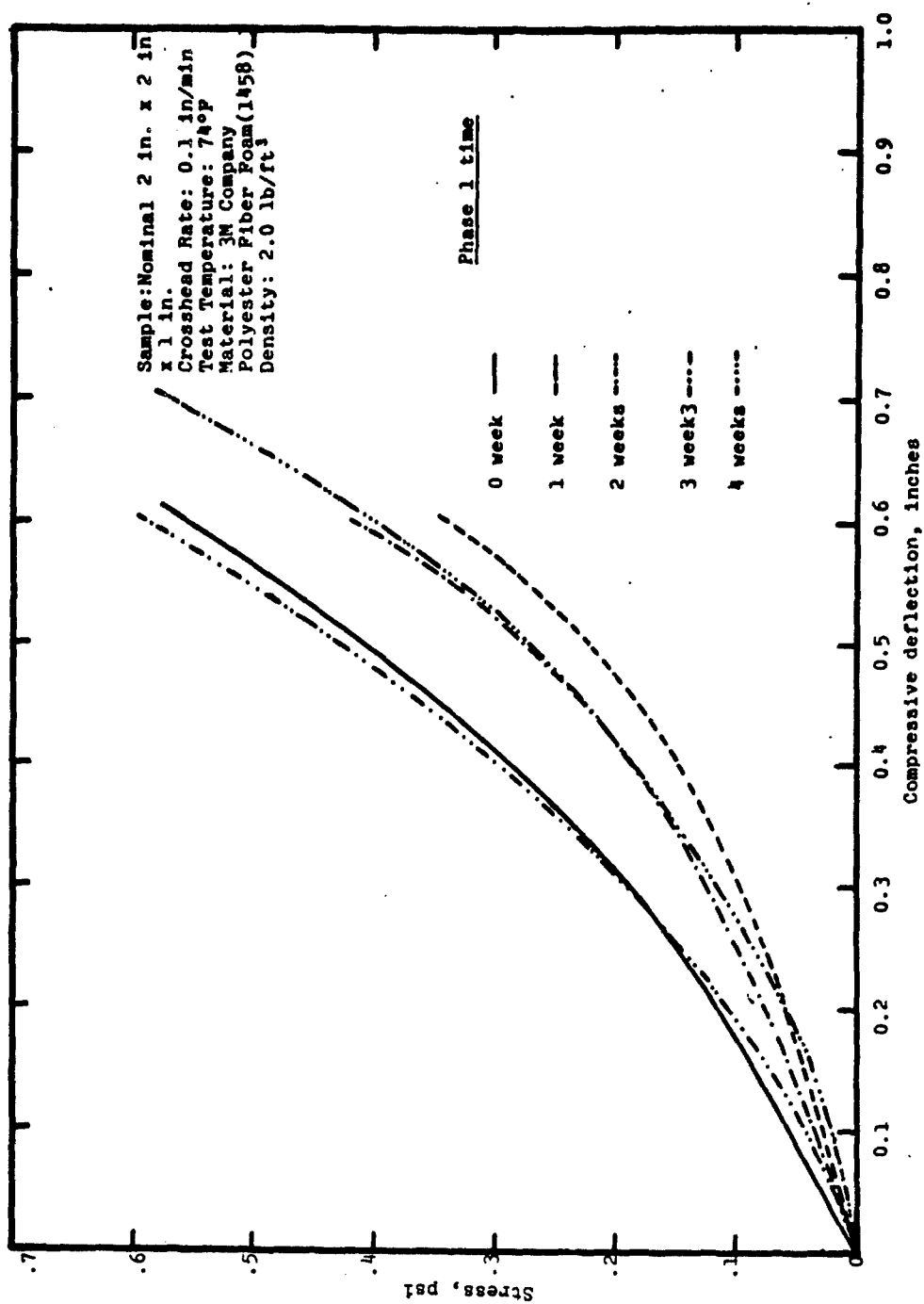


Figure 27. Stress vs Compression of Polyester Fiber Foam Following Phase 1 Exposure for Four Weeks.

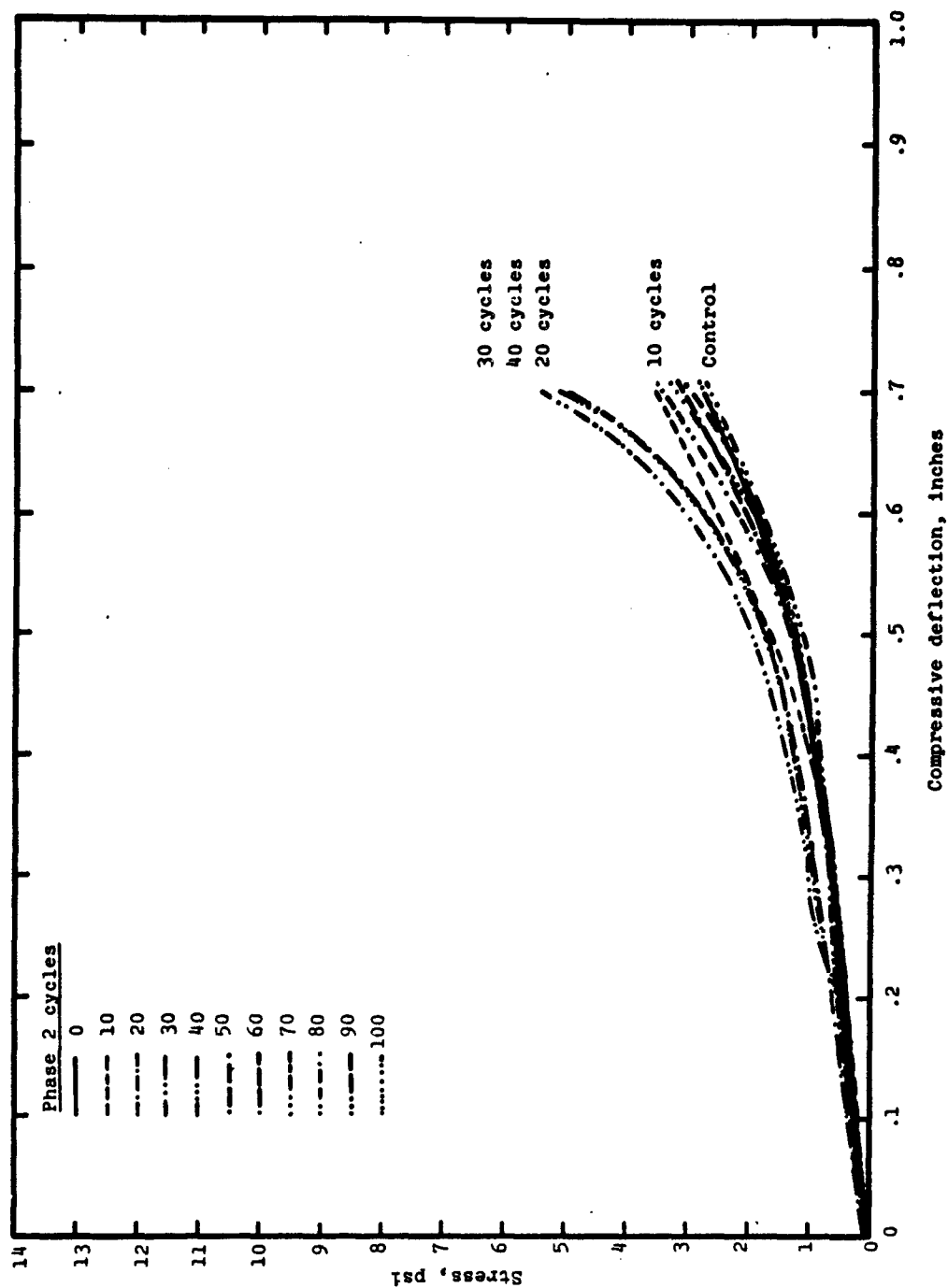


Figure 28. Stress vs Compression of Polyester Fiber Foam Following Phase 2 Exposure for 100 Cycles (ten weeks).

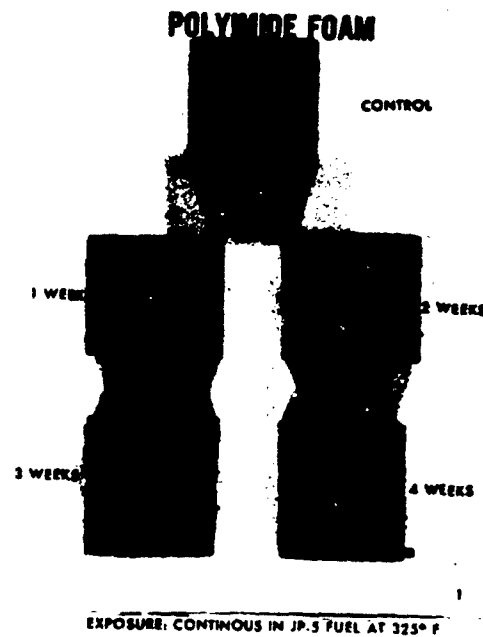
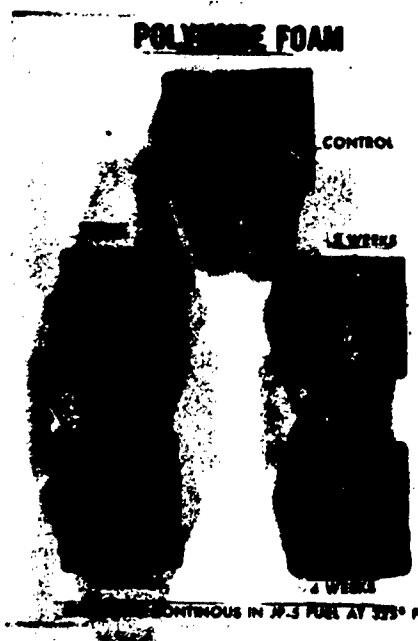
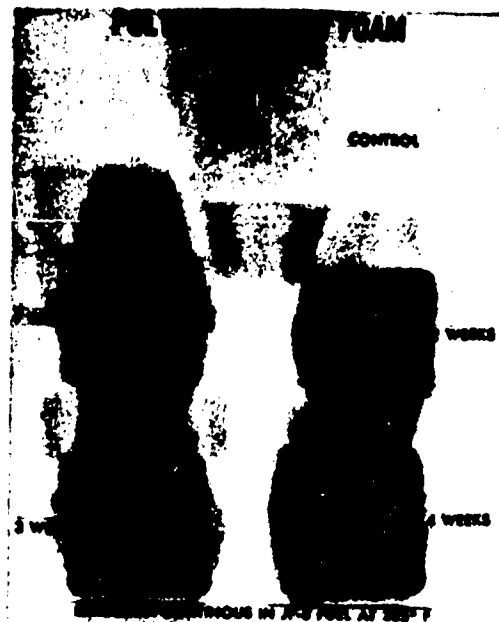


Figure 29. Photographs of High Temperature Foams Following Exposure to JP-5 Fuel at Elevated Temperatures.

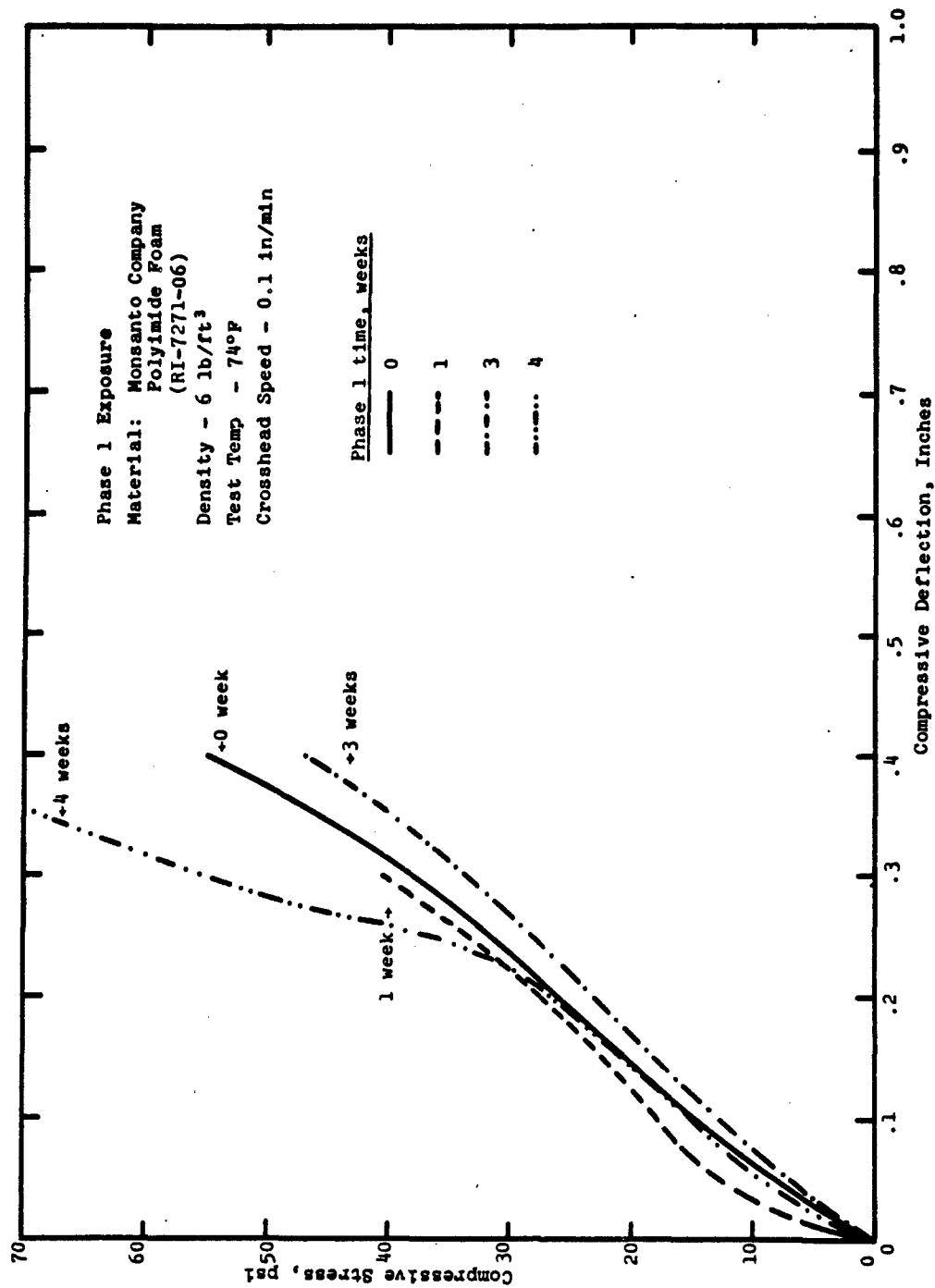


Figure 30. Stress vs Compression for Polyimide Foam Following Phase 1 Exposure for Four Weeks.

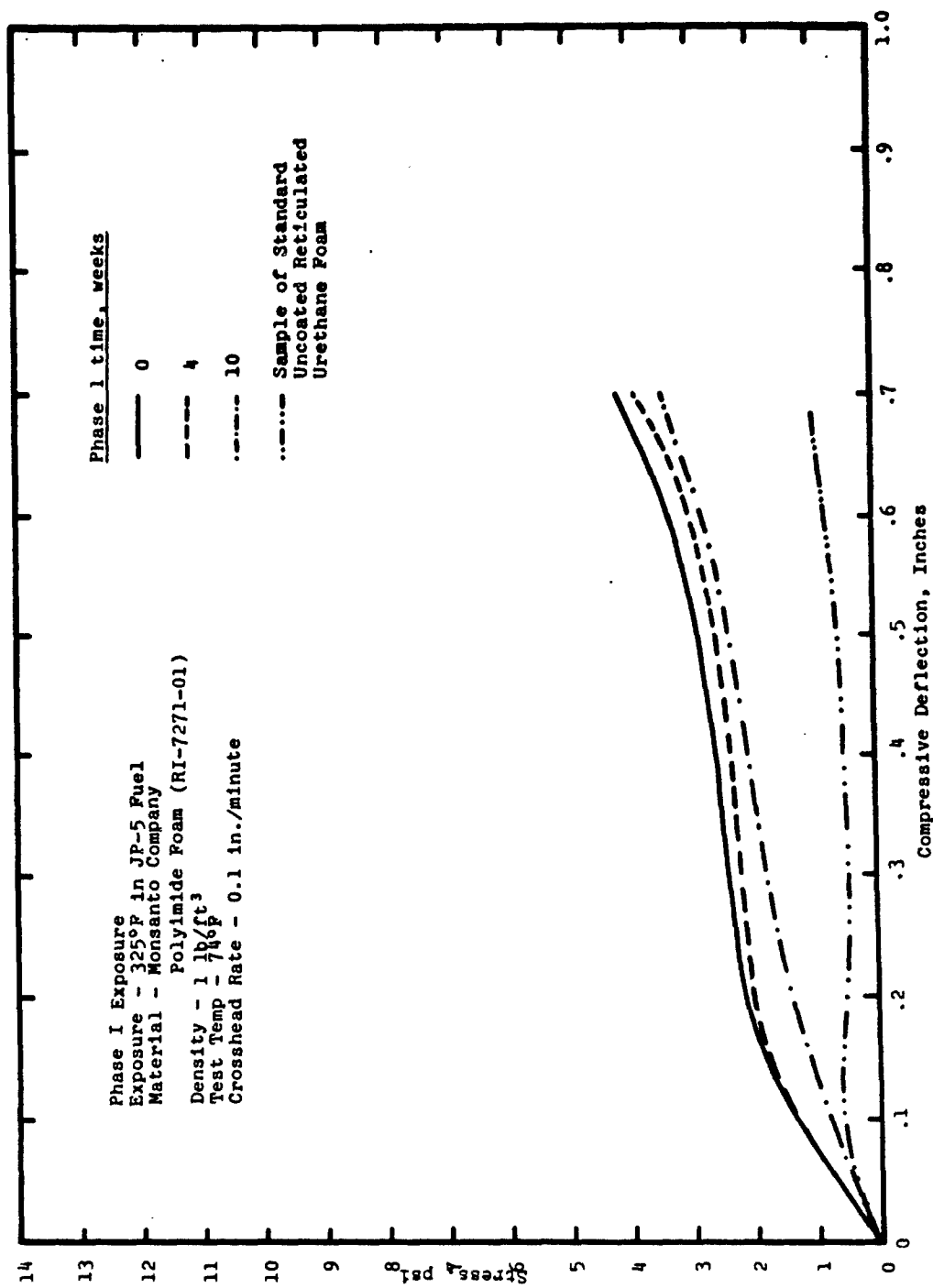


Figure 31. Stress vs Compression for Polyimide Foam Following Phase I Exposure for Ten Weeks.

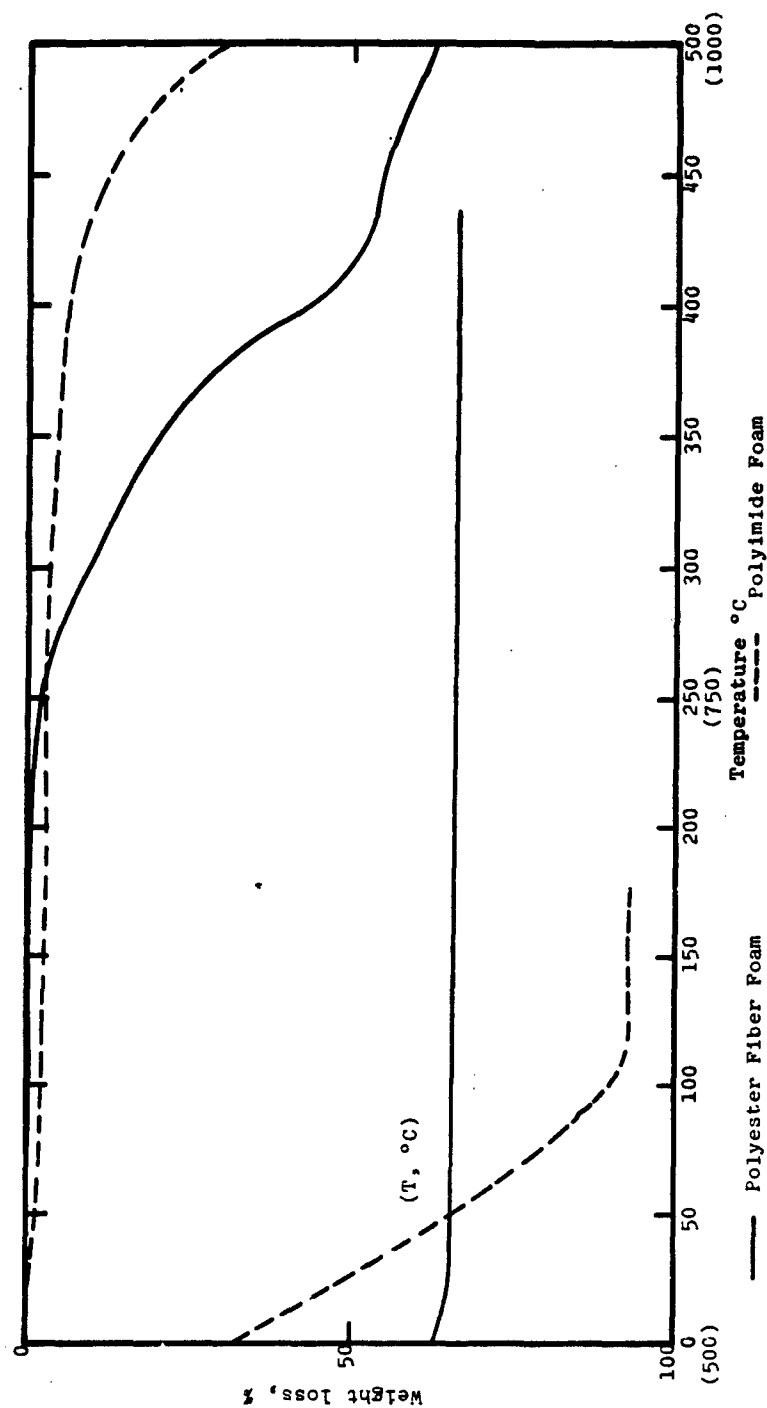


Figure 32. Thermal Gravimetric Analysis of the Polyester and Polyimide Foams in Air.

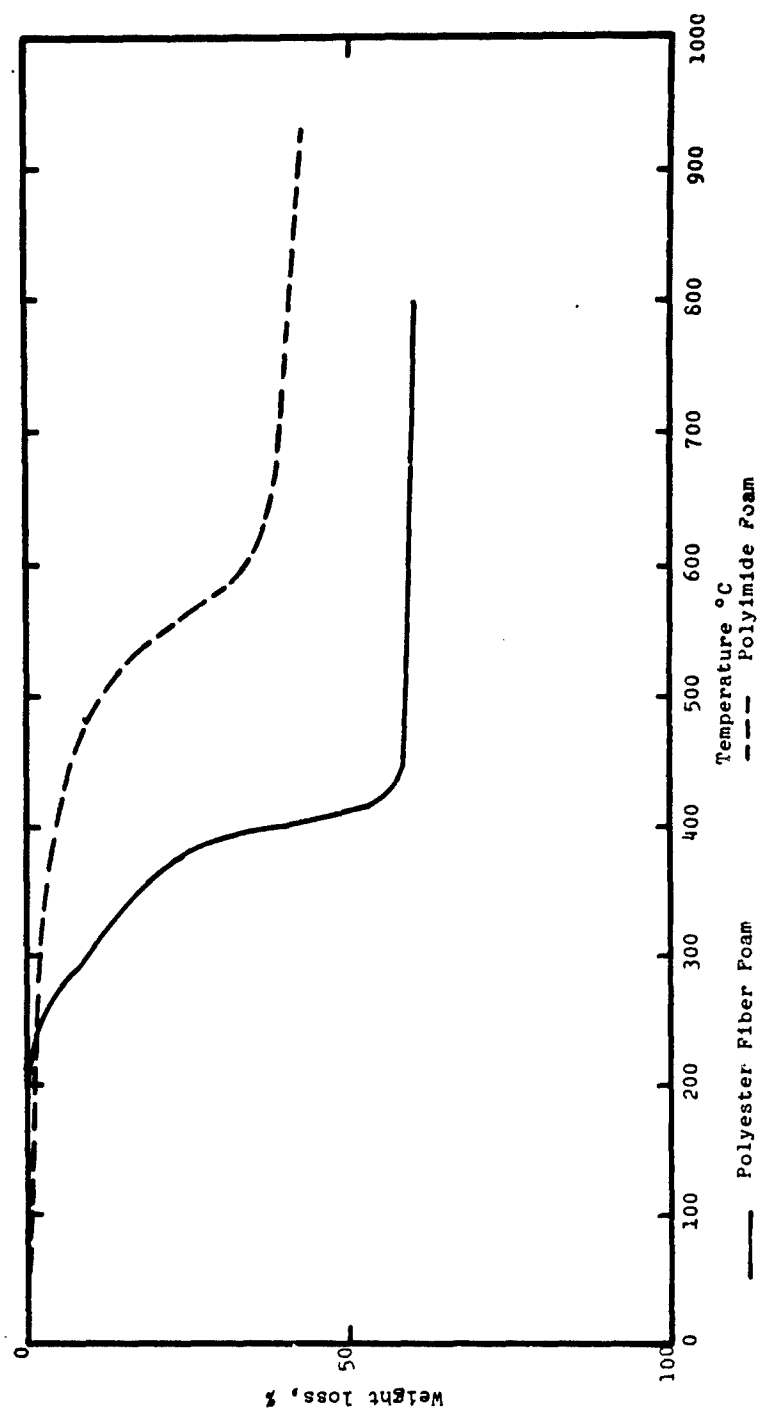


Figure 33. Thermal Gravimetric Analysis of the Polyester and Polyimide Foams in an Inert Atmosphere.



Figure 34. Large Celled Polyimide Foam Prepared
From Polyimide Powders by Salt Ball
Extraction Technique (Density 2.4 lb/ft^3).

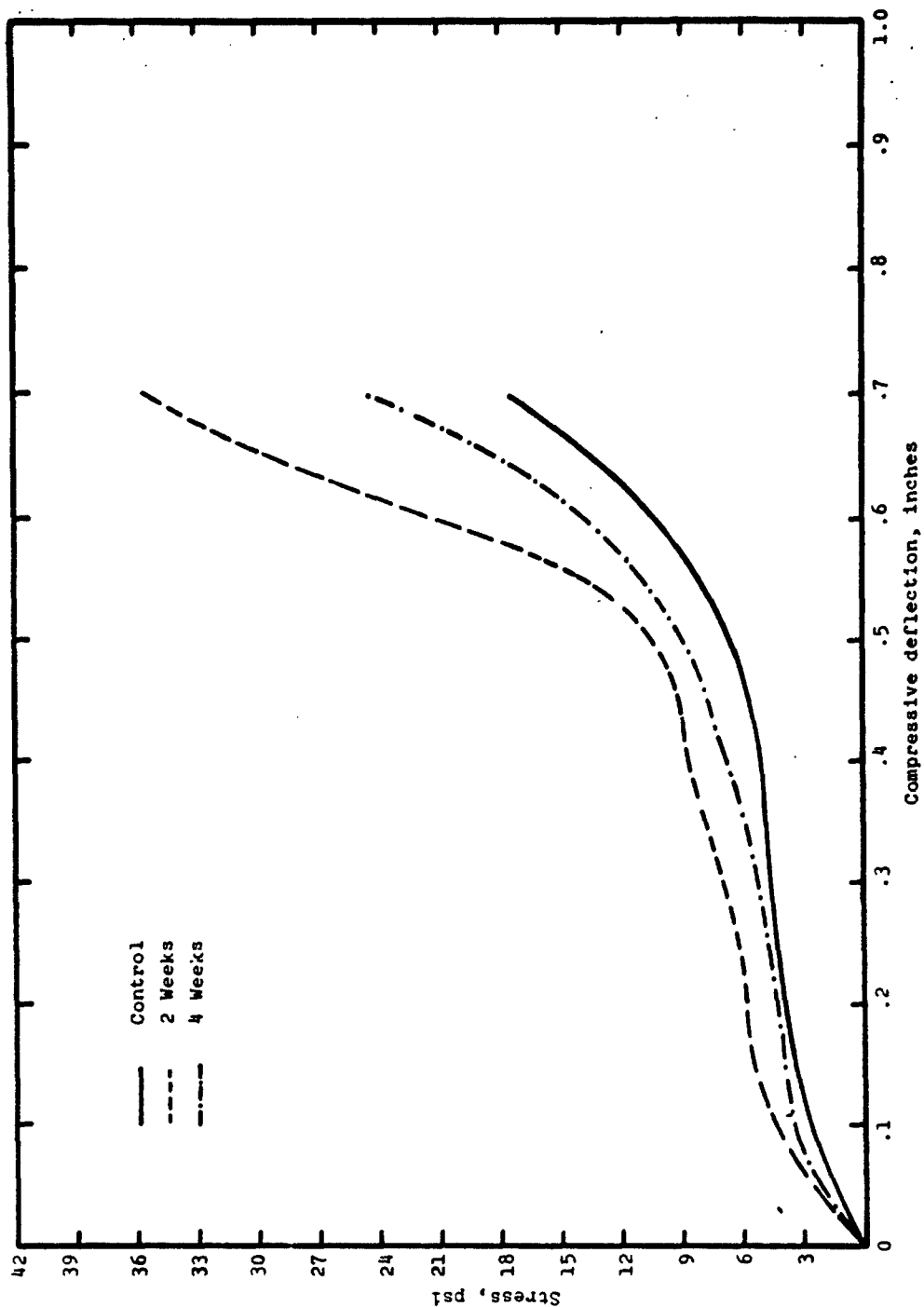


Figure 35. Stress vs Compression for Large Celled Polyimide Foams Following Phase 1 Exposure for Four Weeks.

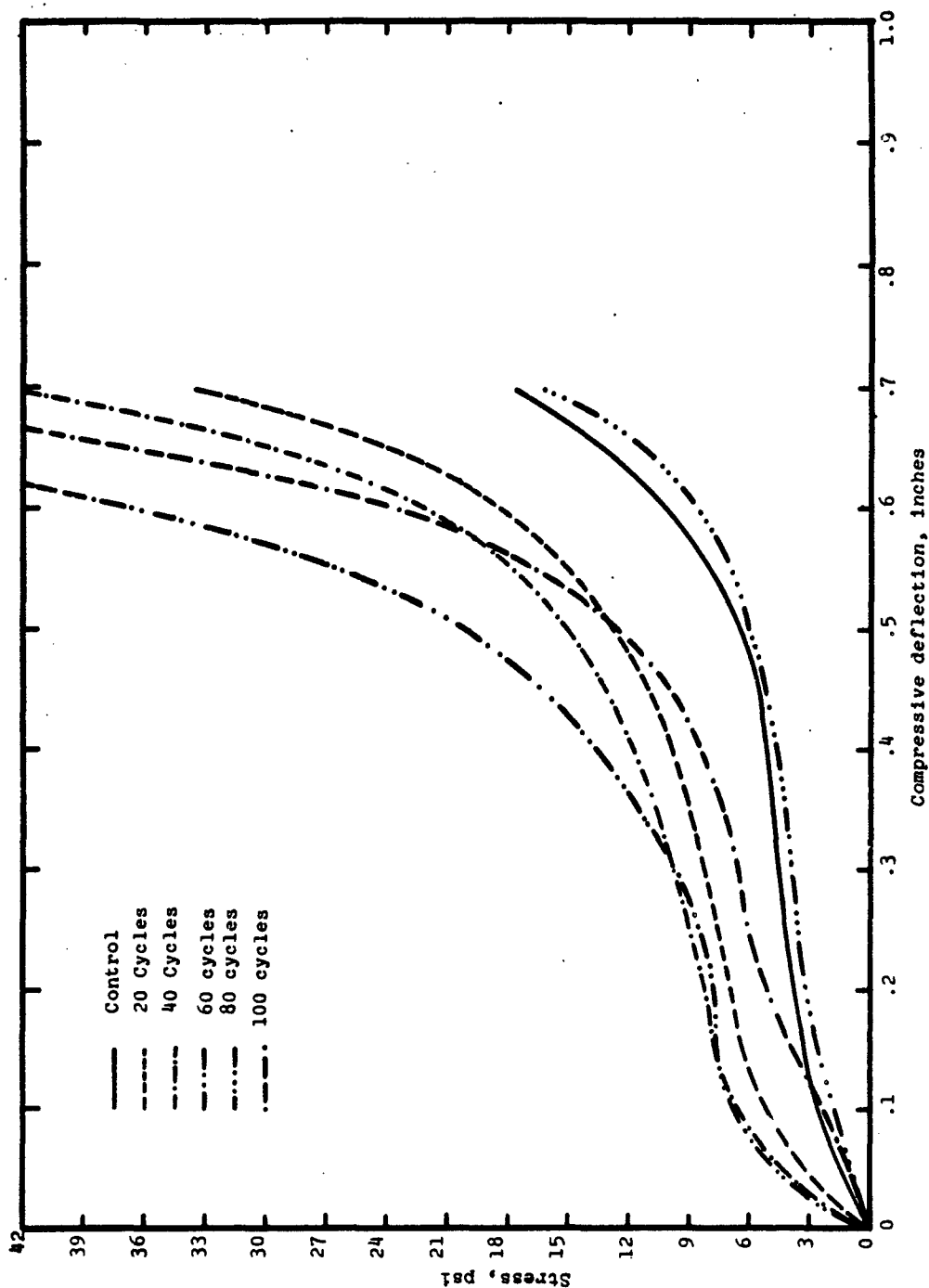


Figure 36. Stress vs Compression for Large Celled Polyimide Foams Following Phase 2 Exposure for 100 Cycles (ten weeks).

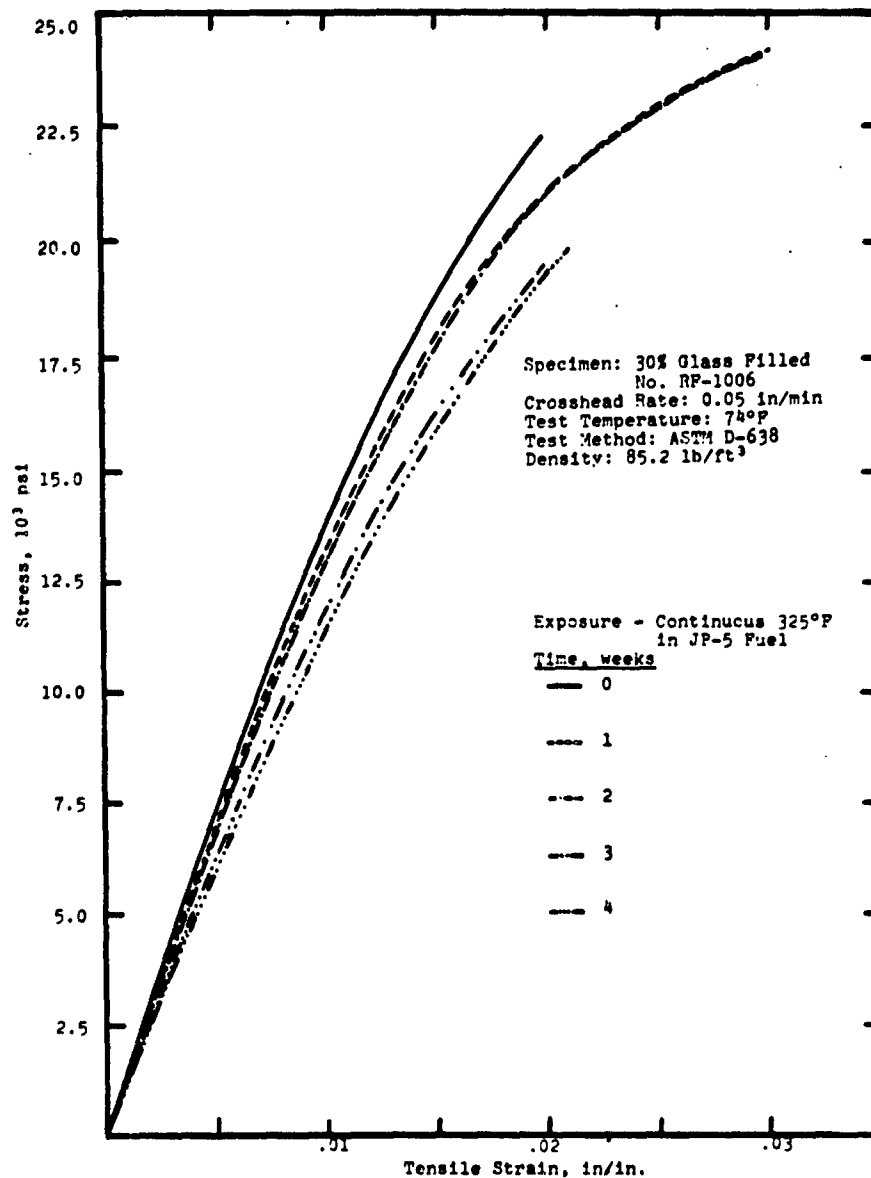


Figure 37. Tensile Stress vs Strain for Glass Filled Nylon 66 Following Phase 1 Exposure for Four Weeks.

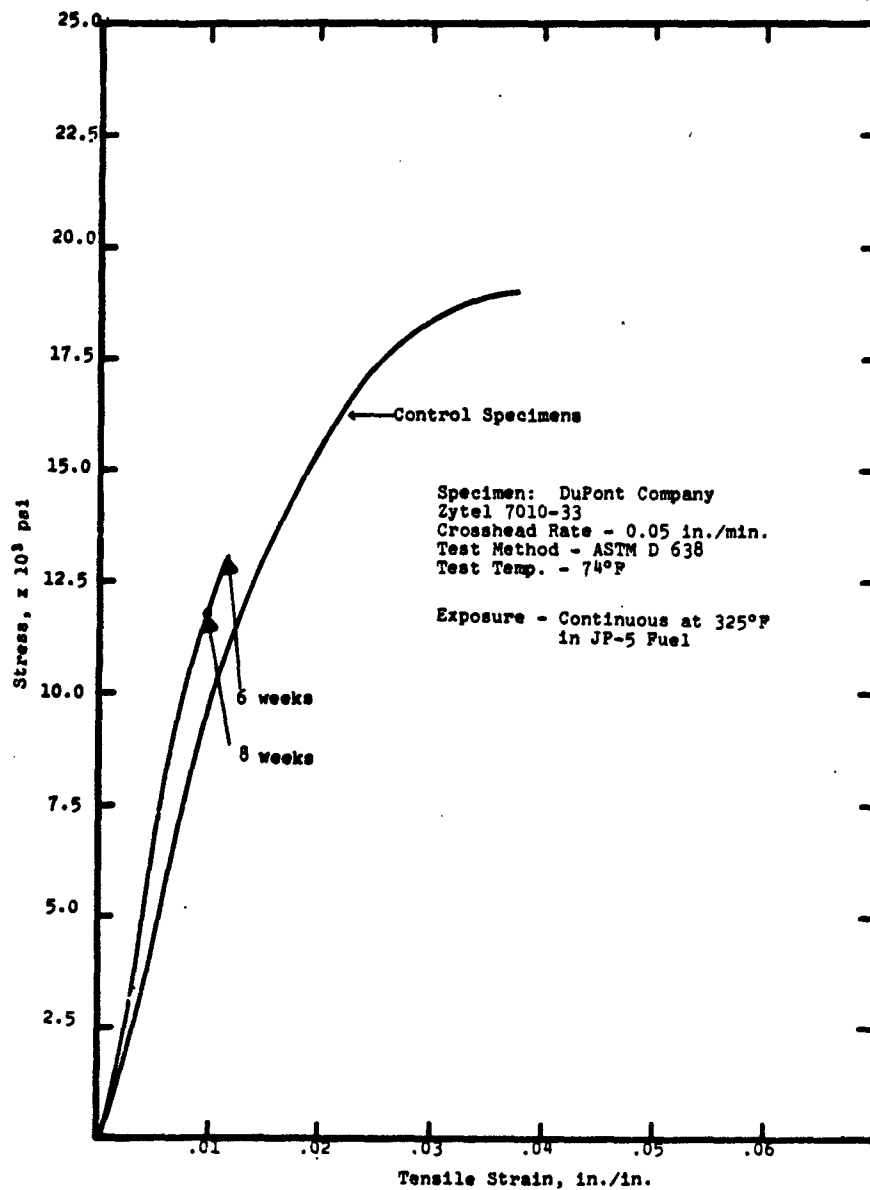


Figure 38. Tensile Stress vs Strain for Glass Filled Nylon 66 Following Phase 1 Exposure for Eight Weeks.

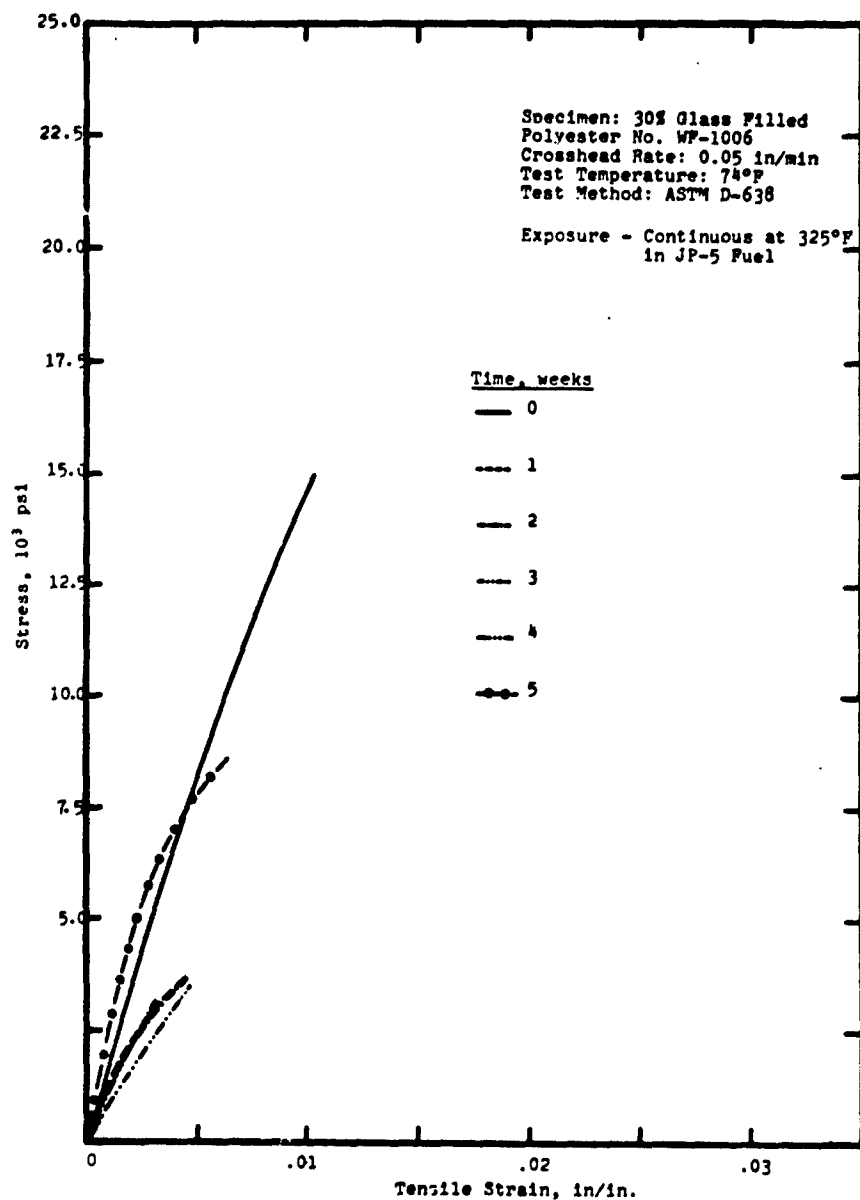


Figure 39. Tensile Stress vs Strain for Glass Filled Polyester Following Phase 1 Exposure for Four Weeks.

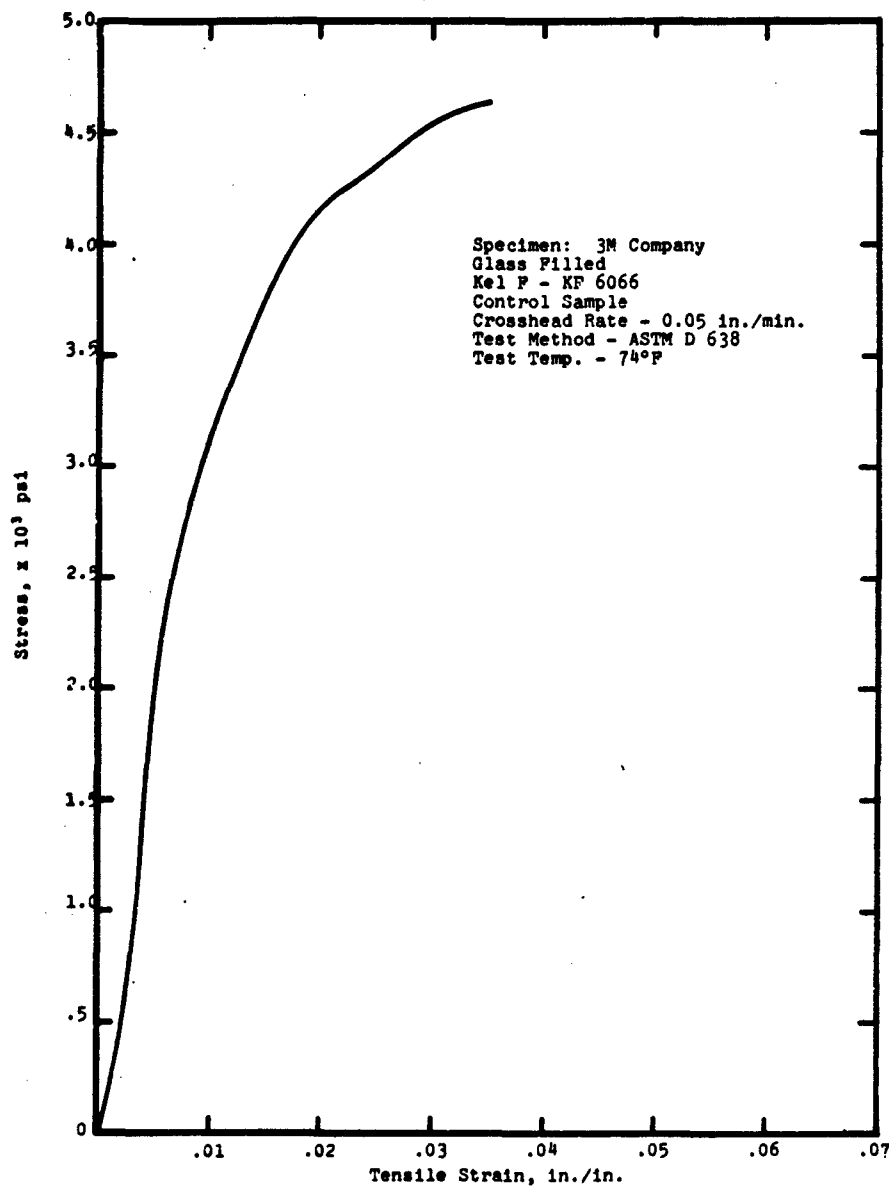


Figure 40. Tensile Stress vs Strain for Unexposed Glass Filled Polychlorotrifluoroethylene.

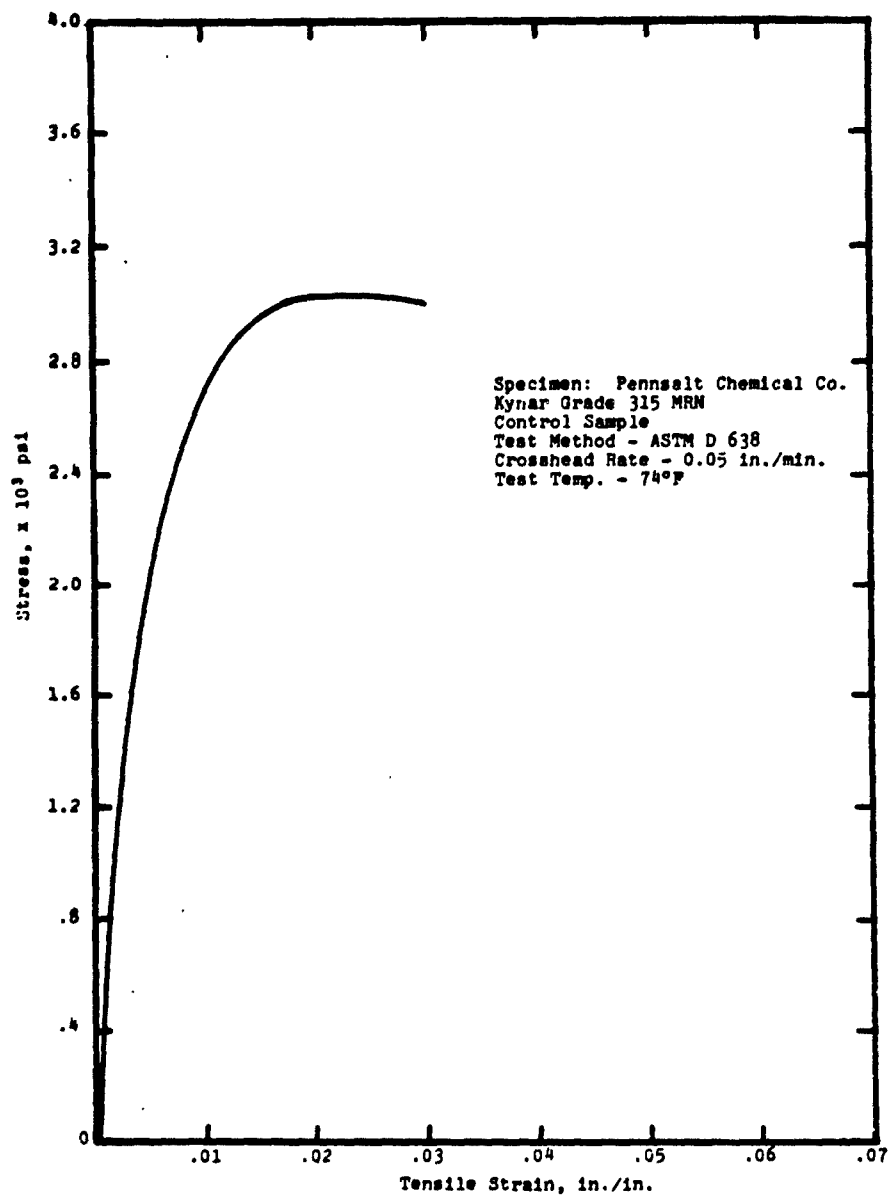


Figure 41. Tensile Stress vs Strain for Unexposed Polyvinylidenechloride.

Reproduced From
Best Available Copy

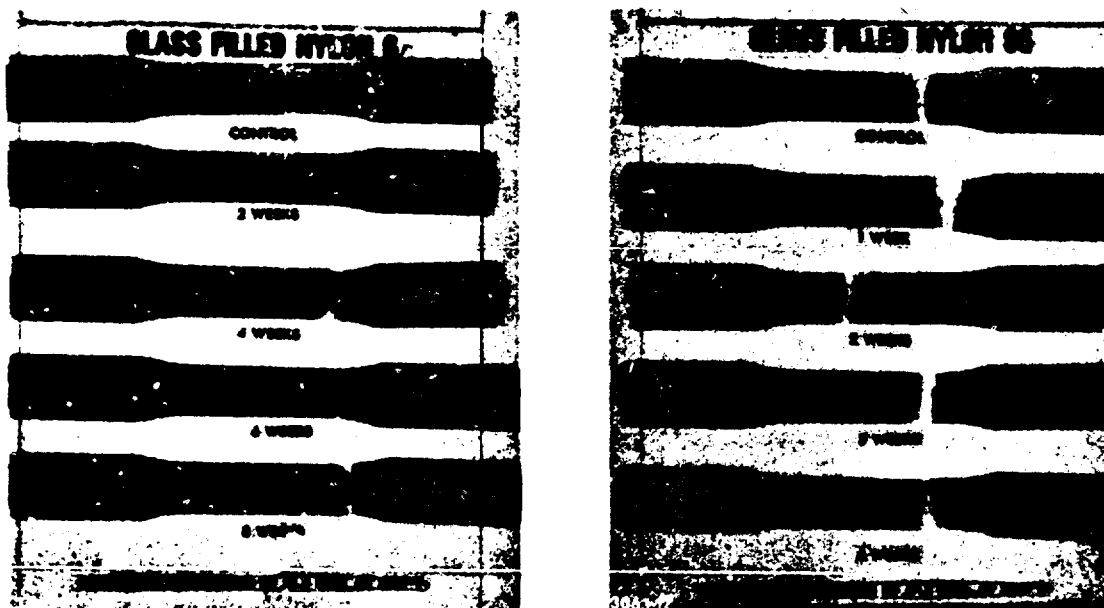


Figure 42. Photographs of Glass Filled Thermoplastics Following Exposure to JP-5 Fuel at Elevated Temperatures.

Reproduced From
Best Available Copy

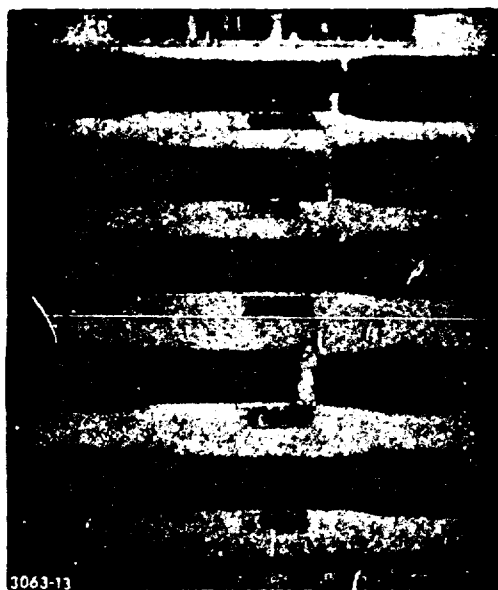


Figure 43. Photographs of Glass Filled Thermoplastics Following Exposure to JP-5 Fuel at Elevated Temperatures.

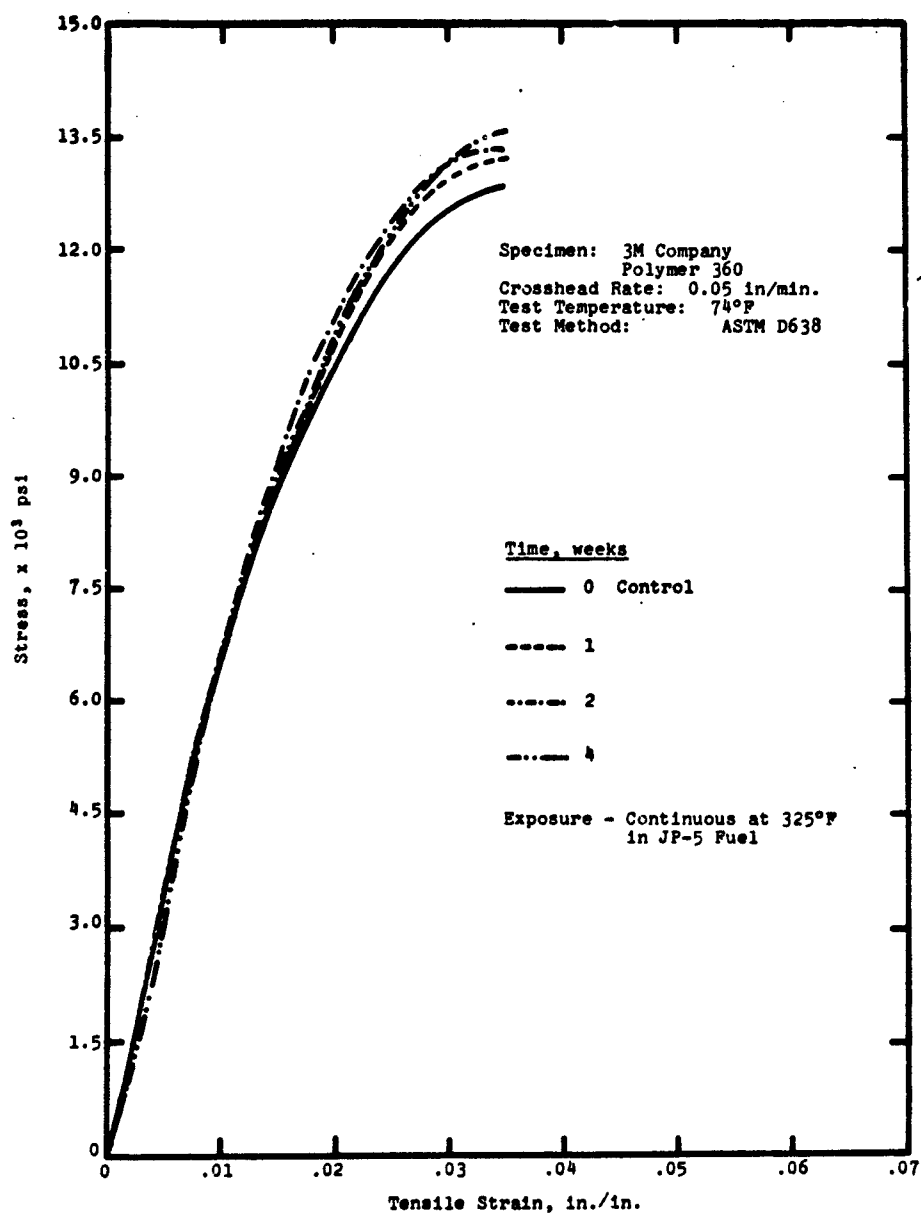


Figure 44. Tensile Stress vs Strain for Polymer 360 Following Phase 1 Exposure for Four Weeks.

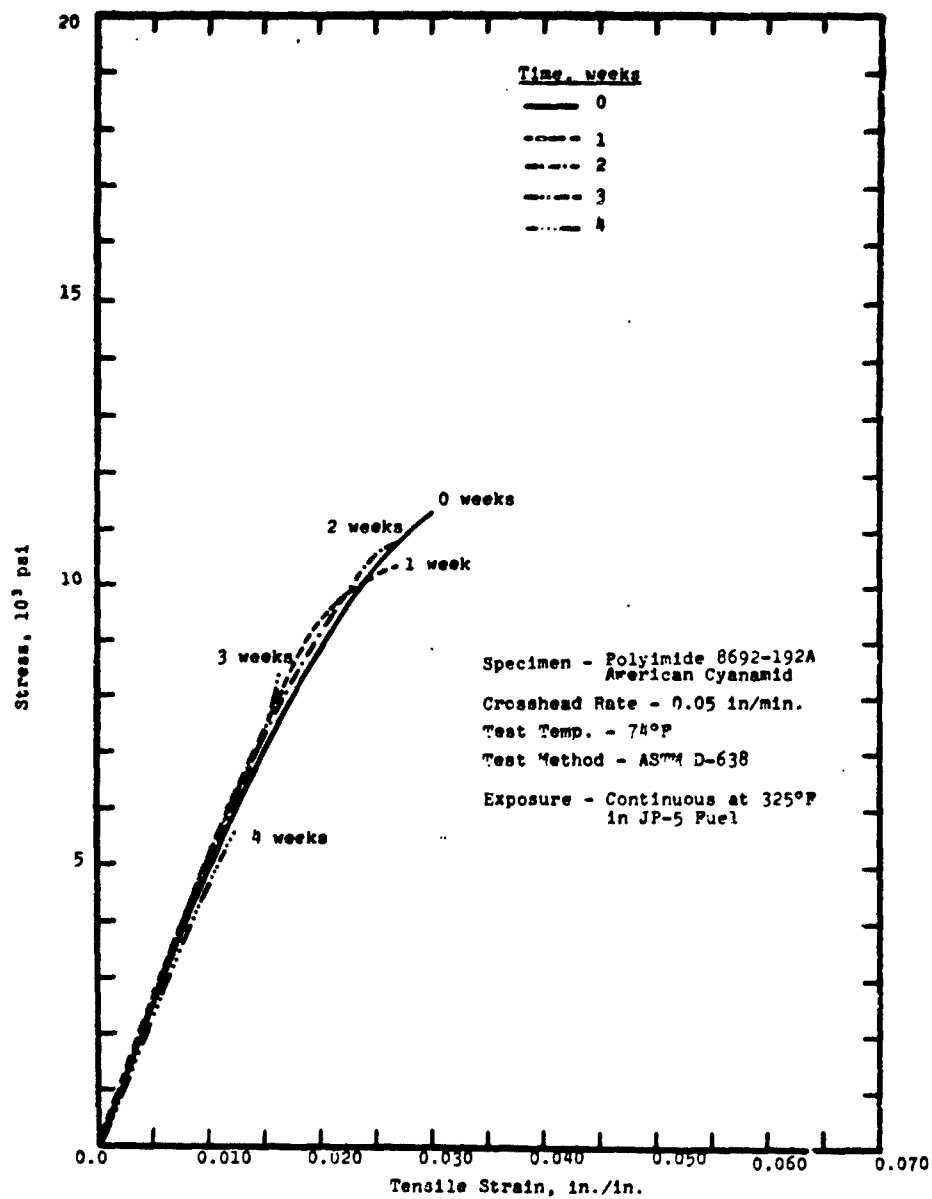


Figure 45. Tensile Stress vs Strain for Thermo-plastic Polyimide Following Phase 1 Exposure for Four Weeks.

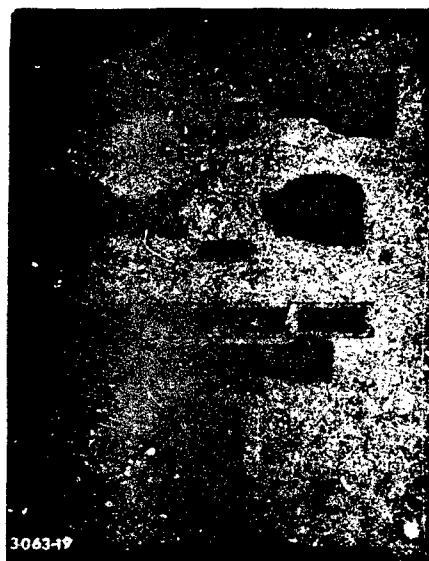
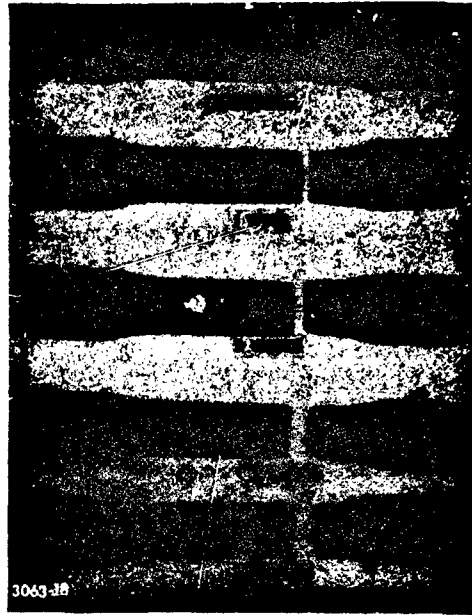
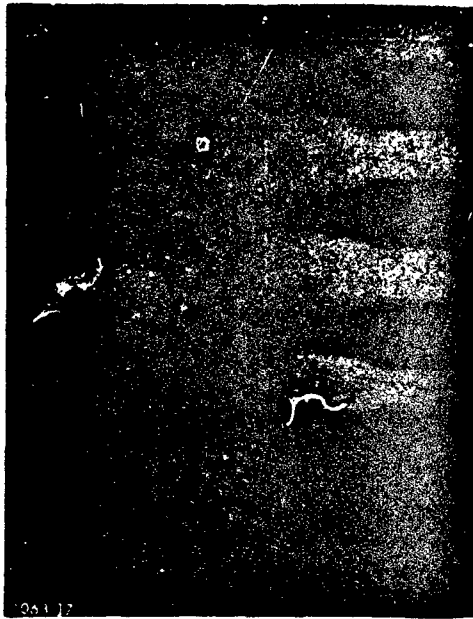


Figure 46. Photographs of Thermoplastic Materials Following Exposure to JP-5 Fuel at Elevated Temperatures.

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Monsanto Research Corporation Dayton Laboratory Dayton, Ohio		2a. REPORT SECURITY CLASSIFICATION Unclass	
		2b. GROUP	
3. REPORT TITLE Evaluation of High Temperature Fire and Explosion Suppressant Configurations for Aircraft Fuel System Applications			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Technical Documentary Report AFML-TR-69-319, 1 July 1968 to 30 Sept 1969			
5. AUTHOR(S) (First name, middle initial, last name) G. L. Ball III A. Wojtowicz I. O. Salyer			
6. REPORT DATE March 1970		7a. TOTAL NO. OF PAGES 62	7b. NO. OF REFS None
8a. CONTRACT OR GRANT NO. F33615-67-C-1716		8b. ORIGINATOR'S REPORT NUMBER(S) MRC-DA-239	
A. PROJECT NO. 7381		9. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
10. DISTRIBUTION STATEMENT This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory (MAAE) Wright-Patterson Air Force Base, Ohio 45433.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY	
13. ABSTRACT <p>A program was conducted for investigation of materials to provide improved thermal resistance in an aviation fuel environment. It was desirable that the compositions obtained be similar to a 10-pore-per-inch reticulated urethane foam used in a fire and explosion suppressant application.</p> <p>Toward providing polymeric structures with improved thermal resistance, four classes of materials were investigated. These were coated foams, uncoated foams, filled thermoplastics, and unfilled thermoplastics. Specific materials were selected and evaluated for their retention of mechanical performance as a function of time following exposure to JP-5 fuel at elevated temperatures.</p> <p>The most promising materials obtained in the program consisted of a polyester fiber mat and a low-density, open-celled polyimide foam. Both of these materials exhibited excellent retention of mechanical strength and modulus for 10-week exposure periods in JP-5 fuel at 325°F.</p> <p>Foams coated with nylon 11, thermoplastic polyimide, and polymer 380 all exhibited some improvement in mechanical performance over their substrate reticulated urethane foam. However, none of these materials proved even partially adequate for the refluxing JP-5 fuel environment.</p> <p>Of the thermoplastics investigated, the nylon 66, polyimide, and polymer 360 all exhibited a substantial retention of mechanical properties in the hyperthermal fuel environment. Of importance was the inclusion of glass fillers, which provided geometrical integrity to the thermoplastic while at the elevated temperatures.</p> <p>In order to establish some utility for the coated urethane foams the mechanical properties of a series of foams were evaluated at temperatures less than 325°F and greater than ambient and a useful temperature of 200°F was established.</p> <p>(This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory (MAAE) Wright-Patterson Air Force Base, Ohio 45433.)</p>			

DD FORM 1473
NOV 65UNCLASSIFIED
Security Classification

Security Classification